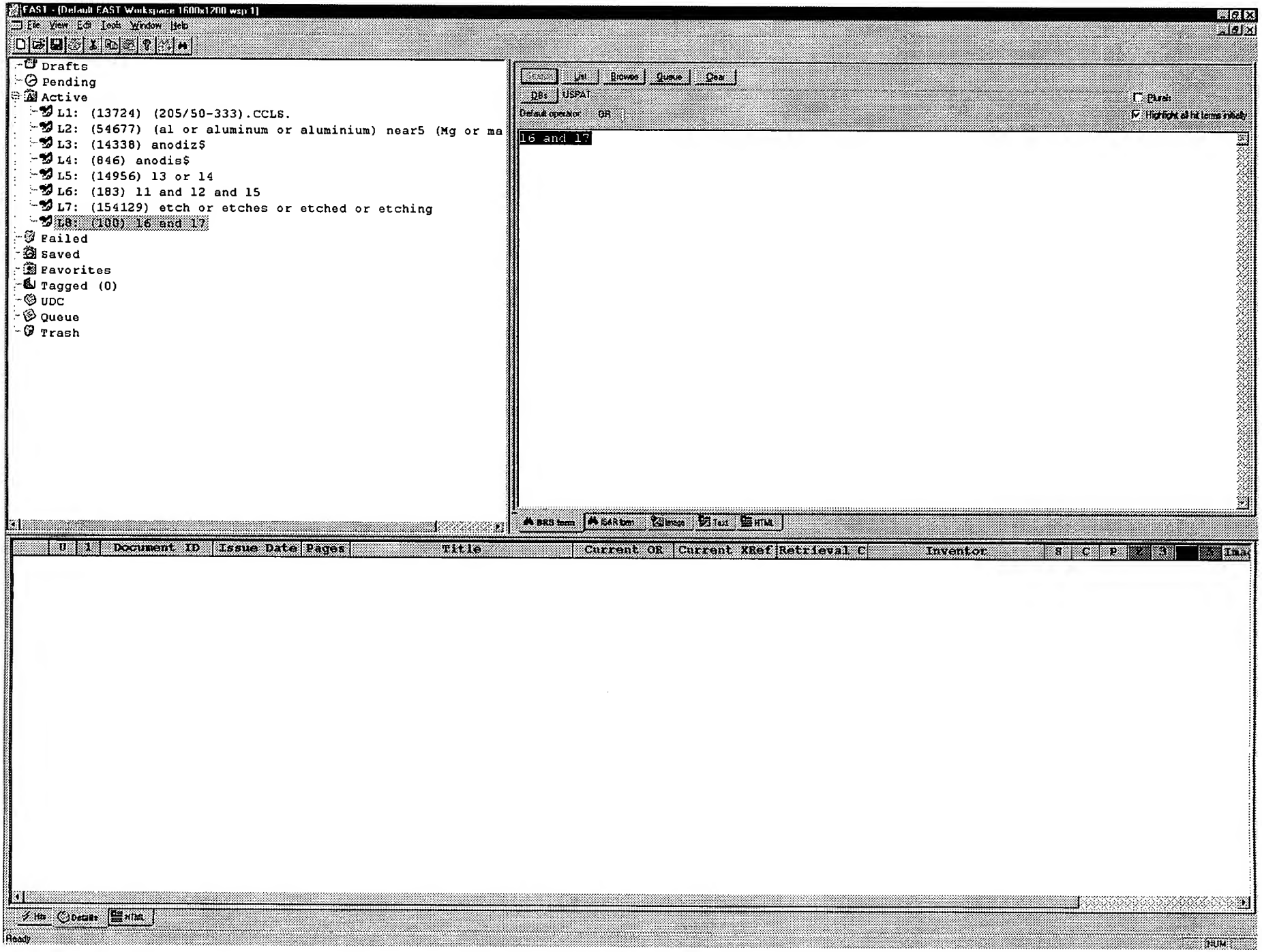
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	Document ID #	Pages	1	2	3	4	5	S	C	P	Kind Codes	Source
11	US 6027629 A	28						☑				USPAT
12	US 6024858 A	20						☑				USPAT
13	US 5873771 A	9						☑				USPAT
14	US 5705225 A	8						☑				USPAT
15	US 5503074 A	11						☑				USPAT
16	US 5496651 A	13						☑				USPAT
17	US 5485294 A	13						☑				USPAT

US-PAT-NO: 5496651

DOCUMENT-IDENTIFIER: US 5496651 A

TITLE: Machine part resistant to rolling friction

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Detailed Description Text - DETX (6):

In Japanese Patent Laid-open (Kokai) No. Hei 4-26792, the inventors of the present invention disclosed facts that the hardness of electrodeposit layers must be Hv 500 or above to provide the machine parts with sufficiently high rolling wear resistance, the surface roughness of the base parts prepared in an Rs of 0.5 .mu.m or above and a PPI.sub.50 of 130 or above by etching or shot blasting provide a strong anchoring effect for firmly anchoring the electrodeposit layer to the surfaces of the base parts of a titanium alloy or an aluminum alloy, a preferable P content of the surfaces of the electrodeposit layers is in the range of about 2 to about 7%, and the gradient distribution of P content in the electrodeposit layers in the direction of thickness of the same improves the rolling wear resistance effectively.

Detailed Description Text - DETX (15):

Gears are used widely in bicycles, automobiles, aircraft and the like for transmitting power. It is essential that gears have high power transmission ability, excellent wear resistance, pitting resistance, flaking resistance and fatigue cracking resistance. Generally, gears are formed of carbon steels and the tooth surfaces of gears are hardened by induction hardening or flame hardening. Some lightweight gears are formed of aluminum alloys. However aluminum alloy gears have poor wear resistance. Although the surfaces of aluminum alloy gears, in general, are anodized to improve the wear resistance, the wear resistance of such anodized aluminum alloy gears is not high enough.

Detailed Description Text - DETX (16):

Al--Cu alloys 2014 and 2017, and Al--Mg alloy 5083 (A.A. Standards) are used for forming gears having sufficiently high strength.

Detailed Description Text - DETX (22):

An anodized layer or a zincate layer formed in the surface of a gear of an aluminum alloy so as to underlie the Ni--P electrodeposit layer further enhances the adhesion of the Ni--P electrodeposit layer to the surface of the gear, because the anodized layer have many pinholes having an anchoring effect to anchor the Ni--P electrodeposit layer firmly thereto, so that the Ni--P electrodeposit layer is difficult to separate from the surface of the gear.

Detailed Description Text - DETX (23):

It is desirable to form an anodized layer or a zincate layer in the surface of the base part of an aluminum alloy before plating the base part with a Ni--P

As shown in FIG. 6, when testing the wear resistance of a test specimen 1, the test specimen 1 was held with the chuck of the rolling wear resistance tester, and tapered evaluation surface 2 of specimen in contact with a plurality of balls 3 retained on a bearing race 4 attached to a rotary shaft 5.

A load P of 100 kgf was applied to the test specimen 1 and the rotary shaft 5 was rotated at 100 rpm. The rolling wear resistance of the test specimen 1 was represented by the cycles of rotation of the rotary shaft 5 in a time from the start of the rotary shaft 5 to a moment when the vibrational acceleration exceeded 0.3 G. Test results and plating conditions are summarized in Table 1.

As is obvious from Table 1, the stresses induced respectively in the Ni—P electrodeposit layers of the test specimens in accordance with the present invention are as low as 10 kgf/mm^2 or below, whereas the stresses induced respectively in the Ni—P electrodeposit layers, which were formed by using plating baths not containing any stress relaxing agent, of the comparative examples are as high as 20 kgf/mm^2 or above. The rolling wear resistance of the Ni—P electrodeposit layers in accordance with the present invention is far higher than that of the Ni—P electrodeposit layers of the comparative examples. The P content and hardness of the Ni—P electrodeposit layers of the present invention are equal to desired values, respectively.

Second Embodiment

Cylinders of different aluminum alloys were subjected to a degreasing process, a first washing process, a chemical etching process, a second washing process and a surface activating process using hydrofluoric acid in that order for pretreatment. Some of the pretreated cylinders were subjected to an anodizing process and the rest were subjected to a zincate layer forming process. Then, the cylinders were coated with Ni—P electrodeposit layers by different electroplating processes, respectively, to obtain test specimens Nos. 1 to 18.

Plating Baths

$NiSO_4.6H_2O$: 200 g/l

$NiCl_2.6H_2O$: 50 g/l

H_3PO_3: 4 to 40 g/l

H_3PO_4: 50 g/l

H_3BO_3: 0.5 to 5 g/l

Saccharin: 0.1 to 1.0 g/l

Temperature: 60° C.±5° C.

pH: 1±0.5

Current density: 5 to 30 A/dm^2

The plating baths were stirred by air during plating and the plating conditions were controlled so that the stress induced in the Ni—P electrodeposit layers is within ±5 kgf/m^2.

The hardness of the Ni—P electrodeposit layers of the test specimens thus obtained were measured, the test specimens were subjected to an wear resistance test, and the surfaces of the test specimens were observed visually after the wear resistance test to evaluate the wear resistance of the Ni—P electrodeposit layers.

The quality of the aluminum alloys, the process conditions of the anodizing process and the zincate layer forming process, the P content, hardness and thickness of the Ni—P electrodeposit layers, and the result of evaluation of the wear resistance of the test specimens are summarized in Table 2, in which test specimens Nos. 1 to 13 are those meeting the

requisite conditions of the present invention and test specimens Nos. 14 to 18 are comparative examples. Although the respective hardnesses of all the test specimens Nos. 1 to 18 are higher than Hv 400, the test specimens Nos. 14 and 15 are inferior in rolling wear resistance, which is inferred to be due to the P content of their Ni—P electrodeposit layers of 8% by weight or above, and the test specimens Nos. 16 to 18 are quite inferior in rolling wear resistance because they are not provided with any Ni—P electrodeposit layer.

The test specimens Nos. 1 to 13 meeting the requisite conditions of the present invention are excellent in rolling wear resistance. The test specimens Nos. 1 to 13, 14 and 15 are excellent in the adhesion of the Ni—P electrodeposit layers to the aluminum alloy cylinders.

Third Embodiment

Cylinders of different titanium alloys formed through a hot forming process, a drawing process and an aging process were subjected to a degreasing process, a first washing process, a chemical etching process using a fluoride, a second washing and a surface activating process in that order for pretreatment. The pretreated cylinders were coated respectively with Ni—P electrodeposit layers by different electroplating processes to obtain test specimens Nos. 1 to 18 in Table 3. The same plating baths as those used for electroplating the test specimens in the second embodiment were used and the electroplating conditions were regulated so that the stress induced in the Ni—P electrodeposit layers is in the range of ±5 kgf/mm^2. During the electroplating process, the plating bath was stirred by air. Test specimens thus obtained were subjected to heat treatment of conditions as shown in Table 3.

The hardness of the Ni—P electrodeposit layers was measured and the test specimens were subjected to rolling wear resistance tests. In the rolling wear resistance tests, the test specimens were held in contact with each other so that the bearing stress, i.e., Hertz's contact pressure, was 200 kgf/mm^2 and the test specimens were rotated at a surface velocity in the range of 60 to 100 m/min and a slip ratio in the range of −70 to +40. The test specimens were lubricated by a mobile oil during rotation. The condition of the surfaces of the test specimens were observed visually after rotating the same by 5×10^7 cycles to evaluate the rolling wear resistance.

Test results are summarized in Table 3, in which the test specimens Nos. 1 to 13 are those meeting the requisite conditions of the present invention and the test specimens Nos. 14 to 18 are comparative examples. Although the respective hardnesses of the Ni—P electrodeposit layers of all the test specimens Nos. 1 to 18 are higher than Hv 400, the test specimens Nos. 14 and 15 are quite inferior in rolling wear resistance, which is inferred to be due to the high P content of 10% by weight of their Ni—P electrodeposit layers, and the test specimens Nos. 16 to 18 are quite inferior in rolling wear resistance because they are not provided with any Ni—P electrodeposit layer.

The test specimens Nos. 1 to 13 meeting the requisite conditions of the present invention are excellent in rolling wear resistance, and the adhesion of the Ni—P electrodeposit layers to the corresponding titanium alloy cylinders of the test specimens Nos. 1 to 15 was satisfactory.

Fourth Embodiment

Coil springs were formed by coiling wires of 3.0 mm in diameter of different titanium alloys through a hot forming process, a wire drawing process and an aging process. The

18 heat treat Mg Al anodize (no etching step disclosed)

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14	US 5705225 A	8						☑				USPAT
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18	US 5364522 A	9						☑				USPAT
19	US 5336341 A	10						☑				USPAT
20	US 5314607 A	8						☑				USPAT

Brief Summary Text - BSTX (15):

Furthermore, the base material of the alloy material on which the anodic oxide film is formed is an aluminum alloy, and this enables various kinds of processing, such as drawing, boring, bending, cutting, and local etching, to be conducted on the alloy material with ease for forming into a desired shape, and then an anodic oxide film is formed on the alloy material. It is thus possible to fabricate infrared radiation elements having a complicated shape which was impossible to form in conventional infrared radiation elements, and hence infrared radiation elements of the present invention has wide practical

Brief Summary Text - BSTX (16):

In another aspect of the present invention, the aluminum alloy contains Mg at an amount of about 0.05 to about 6% by weight in the first aspect of present invention previously described.

Brief Summary Text - BSTX (17):

In a third aspect of present invention, there is provided a process of producing an infrared radiation element, comprising the steps of: (a) heating an aluminum alloy material consisting essentially of about 0.3 to about 4.3 weight % of Mn, balance Al, and impurities for dispersing a precipitate of an Al--Mn intermetallic compound at a density of at a minimum about 1.times.10.sup.5 /mm.sup.3 for a size of about 0.01 .mu.m to about 3 .mu.m; and (b) anodizing the heated aluminum alloy material to form an anodic oxide layer thereon.

Brief Summary Text - BSTX (18):

According to a fourth aspect of the present invention, there is provided a process of producing an infrared radiation element, comprising the steps of: casting a molten alloy at a cooling speed of at least about 5.degree. C./sec to produce an aluminum alloy material, the molten alloy consisting essentially of: about 0.8 to about 3.5 weight % of Mn; balance Al; and impurities; heating the aluminum alloy material at about 300.degree. to about 600.degree. C. for at least about 0.5 hour for dispersing a precipitate of an Al--Mn intermetallic compound at a density of at a minimum about 1.times.10.sub.5 /mm.sup.3 for a size of about 0.01 .mu.m to about 3 .mu.m; and anodizing the heated aluminum alloy material to form an anodic oxide layer thereon.

Brief Summary Text - BSTX (19):

In a fifth aspect of the present invention, a process of producing an infrared radiation element comprises the steps of: casting a molten alloy at a cooling speed at least about 5.degree. C./sec to produce an aluminum alloy material, the molten alloy consisting essentially of: about 0.8 to about 3.5 weight % of Mn; about 0.05 to about 2.0 weight % of Mg; balance Al ; and impurities; heating the aluminum alloy material at about 300.degree. to about 600.degree. C. for at least about 0.5 hour for dispersing a precipitate of an Al--Mn intermetallic compound at a density of at a minimum about 1.times.10.sup.5 /mm.sup.3 for a size of about 0.01 .mu.m to about 3 .mu.m; and anodizing the heated aluminum alloy material to form an anodic oxide layer thereon.

end crystal grains of the alloy become rather coarse. The heating is sufficient if the aluminum alloy is kept at 300° C. at a minimum for at least 0.5 hour. If the heating at a minimum temperature of 300° C. is shorter than 0.5 hour, sufficient black anodic oxide film cannot be obtained after anodization.

EXAMPLE 1

Aluminum alloy plates 1 in thick which contained 0.3 wt. %, 2.0 wt. %, 2.5 wt. %, and 4.3 wt. % of Mn, respectively, were fabricated. The aluminum alloy plates were heated at 400° C. for 12 hours to produce aluminum alloy plates having Al—Mn intermetallic compounds uniformly dispersed in them. According to transmission electron microscope observation, precipitates were 3×10^5/mm^3 to 1×10^7/mm^3 in density for a size of 0.01 to 3 μm. Some of the aluminum alloy plates containing 5 wt. % of Mn were broken during rolling.

Subsequently, the aluminum alloy plates was anodized in a 25 wt. % sulfuric acid bath at 7° C. to thereby produce 5, 10, 15, 20, 30, 40 and 50 μm thick anodic oxide films on them, respectively.

Then, these alloy plates were set in a spectroemissivity measuring equipment, in which they were measured. In infrared radiation emissivity in a wavelength of 6 μm at 80° and 300° C. The results are given in Table 1A.

Thereafter, the aluminum alloy plates were respectively heated at 200°, 250°, 300°, 400° and 500° C. for one hour, and after heating, it was observed as to whether or not cracks had been produced. Although it was observed in 0.3% Mn aluminum alloy plates that slight cracks were produced in the anodic oxide films when the anodic oxide films were relatively thick (50 μm), no cracks were visually observed in the other aluminum alloy plates at specified temperatures. In Table IB, only results after heating at 200° C. for one hour are given.

Comparative Test 1

(1) Aluminum alloy plates 1 mm thick which contained 0.9 wt. %, and 5.0 wt. % of Mn, respectively, were heated and anodized in the same conditions as in Example 1. According to transmission electron microscope observation after heating, for the aluminum alloy plates containing 0.1 wt. % of Mn, precipitates were 2×10^4/mm^3 in density for size of 0.02 to 0.8 μm and whereas for the 5.0 wt. % Mn aluminum alloy plates, precipitates were 3×10^5/mm^3 to 1×10^7/mm^3 in density for a size of 0.01 to 3 μm. Some of the aluminum alloy plates containing 5.0 wt. % of Mn were broken during rolling.

Subsequently, as in Example 1 the aluminum alloy plates were anodized in a 25 wt. % sulfuric acid bath at 7° C. to thereby produce 5, 10, 15, 20, 30, 40 and 50 μm thick anodic oxide films on them, respectively.

Then, these alloy plates were tested in the same manner as in Example 1, and the results are given in Tables 1A and IB.

(2) Aluminum plates 1 mm thick of JIS (Japanese Industrial Standards) A1050 (pure aluminum) were anodized in a 25 wt. % sulfuric acid bath at 7° C. to thereby produce 5, 10, 15, 20, 30, 40 and 50 μm thick anodic oxide films on them, respectively.

Then, as in Example 1 these specimens were measured in infrared radiation emissivity in a wavelength of 6 μm at 80° and 300° C. by the spectroemissivity measuring equipment. The results are given in Table 1A.

Thereafter, the plates were respectively heated at 200°, 250°, 300°, 400° and 500° C. for one hour, and after heating, it was visually inspected as to whether or not cracks had been produced. As a result, it was confirmed that cracks were produced in the anodic oxide films of all the specimens except the 5 μm anodic oxide films. As in Example 1, only results of the specimens heated at 200° C. are given in Table 1.

From Table 1A, it is clear that the JIS A1050 specimens deteriorated in emissivity at 300° C. although they were acceptable at 80° C. On the other hand, specimens which fell within the scope of the present invention exhibited excellent emissivity at both 80° and 300° C. It was noted that 0.3% Mn specimens had been slightly degraded in emissivity as compared to 2.0–4.3% Mn specimens.

Regarding pure aluminum plates of Comparative Test 1, the 200° C.×1 hour heating test revealed that cracks were visually observed in anodic oxide layers of all the specimens except 5 μm anodic oxide specimens. In specimens containing 0.3 to 4.3% by weight of Mn according to the present invention, no cracks were visually observed except that 0.3% Mn specimens which had 50 μm anodic oxide layer had slight cracks produced.

TABLE 1A

Concentration of Mn (wt. %)	Temp. (°C.)	Emissivity (wavelength: 6 μm) Thickness of Anodic oxide (μm) 5	10	15	20	30	40	50
Example 1								
0.3	80	0.62	0.65	0.69	0.70	0.72	0.72	0.73
0.3	300	0.63	0.65	0.65	0.68	0.70	0.70	0.73
2.0	80	0.65	0.72	0.75	0.75	0.78	0.80	0.85
2.0	300	0.65	0.75	0.75	0.80	0.82	0.83	0.85
2.5	80	0.68	0.73	0.75	0.75	0.78	0.80	0.85
2.5	300	0.68	0.73	0.77	0.77	0.82	0.82	0.85
4.3	80	0.66	0.68	0.70	0.72	0.72	0.73	0.73
4.3	300	0.65	0.63	0.70	0.72	0.72	0.73	0.73
Comparative Test 1								
0.1	80	0.58	0.60	0.61	0.62	0.65	0.65	0.67
0.1	300	0.48	0.50	0.52	0.53	0.57	0.60	0.62
5.0	80	0.60	0.62	0.62	0.64	0.67	0.69	0.72
5.0	300	0.53	0.57	0.58	0.60	0.61	0.63	0.65
JIS A 1050	80	0.45	0.51	0.53	0.55	0.56	0.61	0.65
JIS A 1050	300	0.25	0.29	0.36	0.42	0.48	0.52	0.58

18 no heat treatment

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15	US 5503074 A	11						☑				USPAT
16	US 5496651 A	13						☑				USPAT
17	US 5485294 A	13						☑				USPAT
18	US 5364522 A	9						☑				USPAT
19	US 5336341 A	10						☑				USPAT
20	US 5314607 A	8						☑				USPAT
21	US 5304298 A	10						☑				USPAT

A first embodiment of an anodizing apparatus according to the present invention is illustrated in FIG. 1. The apparatus is composed of an anodizing bath 11, an electrode 12, a backing roller 15 and two guide rollers 16,17. The inside of the anodizing bath 11 is formed in a semicylindrical form, and the electrode 12 having a circular arc cross-section is provided on the surface so as to be concentric with the backing roller 15. A inlet passage 13 of an electrolyte solution 14 is provided near the right upper edge of the bath 11. The electrolyte solution flows therefrom to fill the space between the electrode 12 and the backing roller 15, and overflows from the left upper edge into a pit provided on the left side of the bath 11. The backing roller 15 is rotatably provided with a clearance of 20 mm, and most of the under half of the backing roller 15 is dipped in the electrolyte solution 14. An upstream guide roller 16 is provided on the left upper side of the backing roller 15 upstream of electrode 12 and a downstream guide roller 17 is provided on the right upper side downstream of electrode 12. Both guide rollers 16,17 are freely rotatable, and connected to the electrode 12 through a power source (not illustrated). The support 18 of aluminum web is engaged so as to travel from the upstream guide roller 16 to the downstream guide roller 17 around and in contact with the backing roller 15. In this state, the web 18 is started to travel by driving to rotate the backing roller 15, and electric current is supplied from the guide rollers 16,17 to the web 18. The electric current flows from the web 18 to the electrode 12 through the electrolyte solution 14, and at that time, anodized layer is formed on the exposed surface of the web 18.

Detailed Description Text - DETX (21):

An anodizing apparatus of another embodiment of the present invention is illustrated in FIG. 2. This apparatus is the same as that of FIG. 1, except that the guide rollers 16,17 are disposed so as to contact the surface to be anodized of the web 18.

Detailed Description Text - DETX (22):

A JIS 1050 aluminum web 0.15 mm in thickness 1000 mm in width was allowed to travel at 60 m/min, and during traveling, the following treatments were conducted. First, the surface was grained by a rotating nylon brush using pumice water suspension was used as the abrasive material to form a surface roughness of 0.5 .mu.m in center line average height. After washing with water, the surface was etched in 10% sodium hydroxide aqueous solution at 70.degree. C. so that the dissolution quantity of aluminum was 6 g/m.sup.2. After washing with water again, the web was neutralized by passing 30% nitric acid aqueous solution followed by washing with water. Then, electrolytic roughening was conducted in 0.7% nitric acid aqueous solution using rectangular alternating waveform (disclosed in Example of Japanese Patent KOKAI No. 52-77702 at an anode voltage of 13 volts a cathode voltage of 6 volts for 20 seconds, and the surface was washed with 20% sulfric acid aqueous solution and then with water.

Detailed Description Text - DETX (23):

The above roughened aluminum web was anodized using the apparatus shown in FIG. 1 at a traveling speed of the web of 50 m/min, at an electrolytic voltage of 30 V at an electric supply of 1000 kW. The electrolyte solution was 20% sulfuric acid aqueous solution. The surface temperature of the web at the exit of the backing roller 15 was 50.degree. C., and a good anodized layer 1.5 .mu.m in

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United States Patent [19]

Kaneko et al.

[11] Patent Number: 5,314,607

[45] Date of Patent: May 24, 1994

[54] APPARATUS AND METHOD FOR ANODIZING SUPPORTS FOR LITHOGRAPHIC PRINTING PLATE

[75] Inventors: Nobuyoshi Kaneko; Tsutomu Kakei; Akio Uesugi, all of Shizuoka, Japan

[73] Assignee: Fuji Photo Film Co., Ltd., Kanagawa, Japan

[21] Appl. No.: 901,674

[22] Filed: Jun. 22, 1992

[30] Foreign Application Priority Data

Jun. 21, 1991 [JP] Japan 3-150083
Nov. 6, 1991 [JP] Japan 3-289849

[51] Int. Cl.[5] C25D 5/00

[52] U.S. Cl. 205/139; 204/206

[58] Field of Search 204/140, 144.5, 434, 204/206; 205/139, 147

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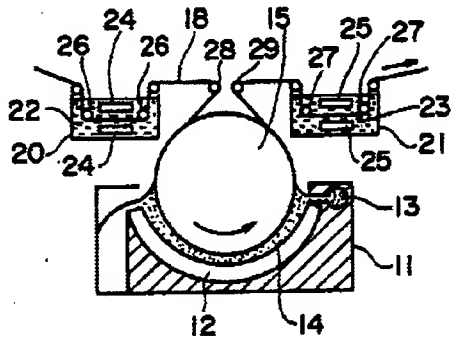
Primary Examiner—John Niebling
Assistant Examiner—Brendan Mee
Attorney, Agent, or Firm—Sughrue, Mion, Zinn, Macpeak & Seas

[57] ABSTRACT

An apparatus for anodizing a support for a lithographic printing plate which comprises a backing roller which guides a web made of aluminum or an alloy thereof in a state of contacting the backing roller, (an) electrode(s) which is/are arranged along the peripheral surface of the backing roller in a concentric arc, an electric supplier which is provided at least on the upstream side or the downstream side of the backing roller, and an electrolyte solution which fills the clearance between the backing roller and the electrode, and an anodizing method using the same. By using the apparatus, the electric voltage loss can be reduced in electrolyte solution, and only a single surface of the supports is anodized without the necessity for providing any special means. Anodizing can be conducted at a high speed and the thickness of anodized layer easily increased. Moreover, thin supports can be anodized without fusion troubles.

8 Claims, 2 Drawing Sheets



a1/1/32 wet anodized pure Al or alloy of Al w.r.t. ... Mg

L8 5000 series contains Mg

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24	US 5264113 A	10											USPAT
25	US 5240589 A	8											USPAT
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29	US 5108812 A	17											USPAT

As previously noted, the present invention is directed to a method of anodizing aluminum or its alloys wherein the anodized surface has low solar absorptivity and high infrared emissivity by use of a step-wise current density and a high bath temperature in the sulfuric acid electrolyte.

Detailed Description Text - DETX (3):

Aluminum or any of its alloys can be anodized according to the invention. These include, for example, the 5,000 series of aluminum alloys containing Mg as a primary alloying element, the 7,000 series containing Zn as primary alloying element, the 2,000 series containing Cu as a primary element and the 6,000 series containing Si and Mg as primary alloying elements. Anodized 5657 aluminum provides a preferred anodized thermal coating for the radiators on spacecraft, and hence 5657 aluminum is the preferred material anodized according to the invention.

Detailed Description Text - DETX (4):

The concentration of the sulfuric acid anodizing electrolyte can range broadly from about 5 to about 25% by weight. Concentrations of sulfuric acid greater than 25% by weight result in an anodic coating giving good optical properties but inferior in terms of ultraviolet radiation stability. Concentrations of sulfuric acid below about 5% by weight are no longer sufficiently conductive and thermally induced electrochemical attack of the sample occurs. A particularly effective and preferred sulfuric acid concentration is 15% by weight.

Detailed Description Text - DETX (5):

During anodizing, the bath temperature of the sulfuric acid electrolyte is maintained at about 30.degree. C. This is considered a high bath temperature for sulfuric acid anodizing, since conventional anodizing in sulfuric acid is usually carried out at room temperature or lower.

Detailed Description Text - DETX (6):

As a feature of the invention anodizing takes place using a direct current step-wise current density procedure at a current density ranging from about 10 to about 20 amperes per square ft. (ASF). The step-wise current density procedure according to the invention proceeds in a manner wherein the first step of the current density procedure is at a higher current density in the current density range, and the last step is at a lower current density in such range. It has been found particularly effective to carry out the anodizing procedure in three steps, namely at 19 ASF, 15 ASF and 10 ASF. At direct current densities above 19 ASF, the samples become too hot and commence to burn. Below about 10 ASF, the required coating thickness is not achieved. Thus the process is quite sensitive to current density.

Detailed Description Text - DETX (7):

It has been found that best results are achieved wherein each of the current density steps of the step-wise current density procedure is maintained constant at that current density for a period of about 20 minutes. Thus a preferred

[54] **PROCESS FOR PRODUCING A HIGH EMITTANCE COATING AND RESULTING ARTICLE**

[75] Inventors: Huong G. Le, Fountain Valley; Dudley L. O'Brien, Los Angeles, both of Calif.

[73] Assignee: McDonnell Douglas Corporation, Long Beach, Calif.

[21] Appl. No.: 876,768

[22] Filed: May 1, 1992

[51] Int. Cl.[5] C25D 11/06

[52] U.S. Cl. 205/328

[58] Field of Search 205/328

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Primary Examiner—T. Tufariello
Attorney, Agent, or Firm—Max Geldin

[57] ABSTRACT

Process for anodizing aluminum or its alloys to obtain a surface particularly having high infrared emittance by anodizing an aluminum or aluminum alloy substrate surface in an aqueous sulfuric acid solution at elevated temperature and by a step-wise current density procedure, followed by sealing the resulting anodized surface. In a preferred embodiment the aluminum or aluminum alloy substrate is first alkaline cleaned and then chemically brightened in an acid bath The resulting cleaned substrate is anodized in a 15% by weight sulfuric acid bath maintained at a temperature of 30° C. Anodizing is carried out by a step-wise current density procedure at 19 amperes per square ft. (ASF) for 20 minutes, 15 ASF for 20 minutes and 10 ASF for 20 minutes. After anodizing the sample is sealed by immersion in water at 200° F. and then air dried. The resulting coating has a high infrared emissivity of about 0.92 and a solar absorptivity of about 0.2, for a 5657 aluminum alloy, and a relatively thick anodic coating of about 1 mil.

17 Claims, No Drawings

19 Al-Mg alloy anodize (no heat treat)

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	Document ID	Pages	1	2	3	4	5	6	7	Kind Codes	Sou
23	US 5266412 A	12						☑			USPAT
24	US 5264113 A	10						☑			USPAT
25	US 5240589 A	8						☑			USPAT
26	US 5217600 A	5						☑			USPAT
27	US 5186767 A	8						☑			USPAT
28	US 5114825 A	9						☑			USPAT
29	US 5108812 A	17						☑			USPAT

US-PAT-NO: 5114825

DOCUMENT-IDENTIFIER: US 5114825 A

TITLE: Substrates for PS plates

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Detailed Description Text - DETX (4):

Moreover, the inventors have also examined influences of other elements of the aluminum alloys and have found that the content of Si should be restricted to not less than 0.05% by weight and less than 0.5% by weight. This is because if it is less than 0.5% by weight, the electrolytic graining treatment is liable to remain unetched portions on the surface of such aluminum alloy plates. Preferably, uniform etching patterns can be obtained if the content of Si is controlled to not less than 0.2% by weight. On the other hand, if it exceeds 0.5% by weight, the electrolytic graining treatment frequently provides non-uniform grained surface.

Detailed Description Text - DETX (8):

Aluminum alloys may further comprise not more than 1.3% by weight of Mg. Mg is added to these alloys to improve the strength thereof without exerting any adverse influences on the electrolytic graining. Most of Mg is occluded in the Al phase to increase the strength thereof, but if the content thereof exceeds 1.3% by weight, the rolling properties of the alloys are lowered and the use of Mg in excess makes the surface of the alloys electrolytically grained non-uniform.

Detailed Description Text - DETX (12):

Prior to electrolytic graining, the aluminum plates is subjected to a surface treatment for cleaning the surface thereof such as removal of rolling oils adhered to the aluminum surface or the abrasive compounds which bite into the surface (if the surface is subjected to mechanical graining). Generally, solvents such as trichloroethylene or surfactants are used to remove the rolling oils to thus make the surface clean. Alternatively, in order to remove both rolling oils and abrasive compounds biting into the surface, there are generally used methods which comprise dipping an aluminum alloy plate in an aqueous solution such as 1 to 3% aqueous solutions of sodium hydroxide, potassium hydroxide, sodium carbonate and sodium silicate at a temperature of 20.degree. to 80.degree. C. for 5 to 250 seconds and then dipping it in 10 to 30% aqueous solution of nitric acid or sulfuric acid at a temperature of 20.degree. to 70.degree. C. for 5 to 250 seconds to perform neutralization and removal of smuts after the alkali etching.

Detailed Description Text - DETX (19):

After desmutting the surface, the aluminum alloy plates are enodized. The anodization may be carried out in a conventionally well known manner, but most useful electrolyte is sulfuric acid. Secondary preferred electrolyte is phosphoric acid. Moreover, the method using a mixed acid of sulfuric acid and

United States Patent [19]

Takizawa et al.

[11] **Patent Number: 5,114,825**

[45] **Date of Patent: May 19, 1992**

[54] **SUBSTRATES FOR PS PLATES**

[75] Inventors: **Kazuhige Takizawa; Hirokazu Sakaki**, both of Shizuoka, Japan

[73] Assignee: **Fuji Photo Film Co., Ltd.**, Minami-Ashigari, Japan

[21] Appl. No.: **361,430**

[22] Filed: **Jun. 5, 1989**

[30] **Foreign Application Priority Data**

Jun. 6, 1988 [JP] Japan 63-138675

[51] Int. Cl.[5] G03C 1/82; G03C 1/493; C25F 3/04

[52] U.S. Cl. 430/159; 430/157; 430/275; 428/469; 204/129.1; 204/129.4; 204/129.75; 205/201; 205/214; 205/325

[58] Field of Search 204/129.1, 129.4, 129.43, 204/33, 129.75, 27, 38.3; 428/469; 430/278, 157, 159

[56] **References Cited**

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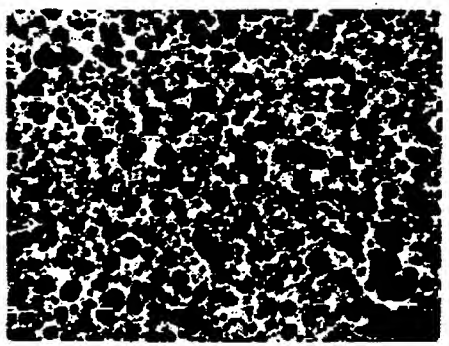
Primary Examiner—Donald R. Valentine
Attorney, Agent, or Firm—Burns, Doane, Swecker & Mathis

[57] **ABSTRACT**

An aluminum alloy substrate for presensitized plates for use in making lithographic printing plates comprises an aluminum alloy plate composed of not less than 0.05% by weight and less than 0.5% by weight of Si; 0.2 to 0.7% by weight of Fe; 0.3 to 1.5% by weight of Mn; less than 0.3% by weight of Cu; and the balance of aluminum and unavoidable impurities, the surface of the aluminum alloy plate being subjected to electrolytic graining treatment. The aluminum alloy substrates for PS plates are favorable for appropriate electrolytic graining treatment and show good printing properties and sufficient strength suitable for high-speed printing operation.

16 Claims, 1 Drawing Sheet



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45	US 4614570 A	12						☑				USPAT
46	US 4601796 A	7						☑				USPAT
47	US 4578156 A	8						☑				USPAT

Detailed Description Text - DETX (2):

In accordance with the invention, an improved highly reflective semi-specular anodized aluminum material is produced from an aluminum alloy which contains essentially only magnesium as an alloying additive. The alloy is either conventionally bright rolled at the plant or else is first mechanically finished or polished to provide a smooth surface. Optionally, the material may then be treated in a brightening bath which may comprise a chemical brightener or an electro brightener. When a chemical brightening step is used, it may also be desirable to subsequently etch the brightened surface in a phosphoric acid etch . The polished and brightened aluminum surface is then anodized in accordance with the invention to provide the desired highly reflective semi-specular surface.

Detailed Description Text - DETX (4):

The aluminum alloy used in accordance with the invention consists essentially of from 0.25 to 1.5 wt. % magnesium and the balance aluminum . No manganese or copper is added as an alloying additive. The maximum amount of copper, manganese, iron and silicon which may be tolerated as impurities is no more than 0.05 wt. % copper, no more than 0.01 wt. % manganese and no more than 0.50 wt. % of either iron or silicon. Preferably, the aluminum alloy used in accordance with the invention consists essentially of from 0.65 to 0.80 wt. % magnesium with the balance aluminum and no more than from 0.10 to 0.20 wt. % iron, 0.07 to 0.13 wt. % silicon, 0 to 0.01 wt. % copper and 0 to 0.01 wt. % manganese present as impurities.

Detailed Description Text - DETX (5):

The aluminum alloy material used to form the highly reflective product may comprise as-rolled sheet or may be subjected to any conventional mechanical polishing techniques as are well known to those skilled in the art. As stated above, if desired the aluminum material may be subjected to a conventional chemical brightening step. However, it has been found that the highly reflective anodized aluminum alloy product of the invention may be formed with only a mechanical bright rolling step prior to anodizing . If the chemical brightening step is used, it may comprise a chemical brightener, such as the Alcoa 5 chemical brightening which comprises the use of a hot mixture of 85% phosphoric acid and 70% nitric acid which is initially mixed in a 19:1 volumetric ratio, although this ratio will change during use due to accumulation of aluminum phosphate in the solution. If a chemical brightening step is used, it may be desirable to subsequently etch the brightened surface in a 30-40% phosphoric acid etch for from 1/4 to 1 minute to insure formation of the desired semi-specular finish.

Detailed Description Text - DETX (6):

The aluminum surface, brightened by either bright rolling or chemical brightening, is then anodized to provide a protective layer of aluminum oxide over the brightened aluminum surface. In accordance with one aspect of the invention, a sulfuric acid anodizing bath is used having a concentration of from 26 to 32 wt. % sulfuric acid, preferably 28 to 32 wt. % sulfuric acid. The temperature of the bath during anodizing is maintained, in accordance with the invention, at from about 60 degrees to 84 degrees F., preferably

United States Patent [19]

Powers et al.

[11] **Patent Number: 4,601,796**

[45] **Date of Patent: Jul. 22, 1986**

[54] **HIGH REFLECTANCE SEMI-SPECULAR ANODIZED ALUMINUM ALLOY PRODUCT AND METHOD OF FORMING SAME**

[75] Inventors: John H. Powers, Lower Burrell; Hang T. Dang, Plum Boro, both of Pa.

[73] Assignee: Aluminum Company of America, Pittsburgh, Pa.

[21] Appl. No.: 784,573

[22] Filed: Jul. 15, 1985

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 651,912, Sep. 19, 1984, which is a continuation of Ser. No. 590,523, Mar. 16, 1984, Pat. No. 4,483,750.

[51] Int. Cl.[4] C25D 5/44

[52] U.S. Cl. 204/33; 204/29; 204/58

[58] Field of Search 204/58, 29, 33

[56] **References Cited**

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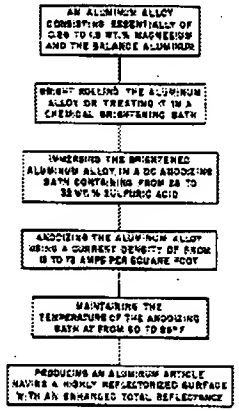
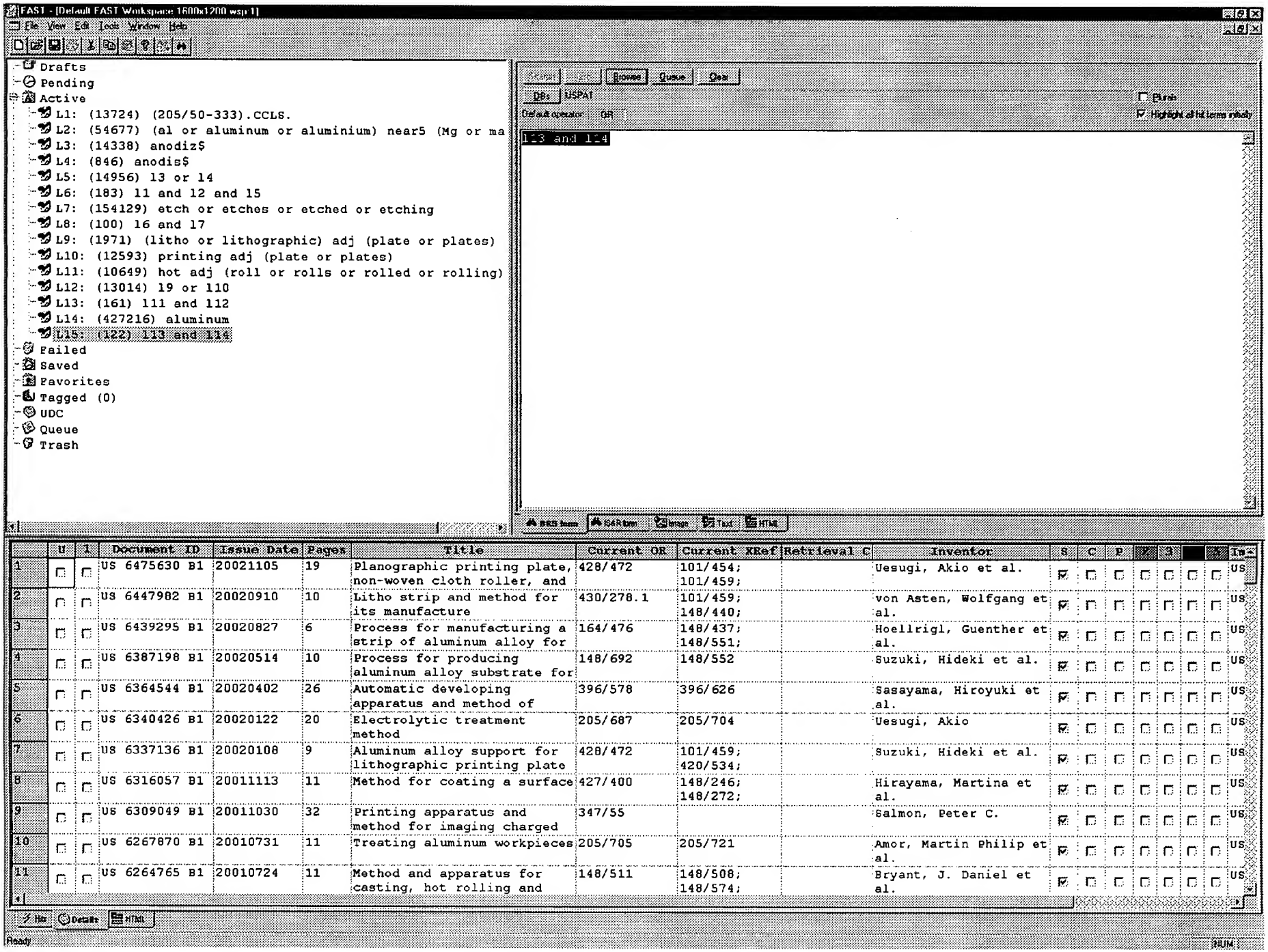
Primary Examiner—R. L. Andrews
Attorney, Agent, or Firm—Andrew Alexander; John P. Taylor

[57] **ABSTRACT**

The invention comprises a highly reflective anodized aluminum alloy product consisting essentially of 0.25 to 1.5 wt. % magnesium with the balance aluminum anodized in a DC anodizing bath containing at least 26% sulfuric acid at a current density of at least 18 amperes per square foot at a temperature of at least 60° F.

23 Claims, 2 Drawing Figures





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1	☐	☐	US 6475630 B1	20021105	19	Planographic printing plate, non-woven cloth roller, and	428/472	101/454; 101/459;		Uesugi, Akio et al.	☑	☐	☐	☐	☐	☐	☐	US
2	☐	☐	US 6447982 B1	20020910	10	Litho strip and method for its manufacture	430/278.1	101/459; 148/440;		von Asten, Wolfgang et al.	☑	☐	☐	☐	☐	☐	☐	US
3	☐	☐	US 6439295 B1	20020827	6	Process for manufacturing a strip of aluminum alloy for	164/476	148/437; 148/551;		Hoellrigl, Guenther et al.	☑	☐	☐	☐	☐	☐	☐	US
4	☐	☐	US 6387198 B1	20020514	10	Process for producing aluminum alloy substrate for	148/692	148/552		Suzuki, Hideki et al.	☑	☐	☐	☐	☐	☐	☐	US
5	☐	☐	US 6364544 B1	20020402	26	Automatic developing apparatus and method of	396/578	396/626		Sasayama, Hiroyuki et al.	☑	☐	☐	☐	☐	☐	☐	US
6	☐	☐	US 6340426 B1	20020122	20	Electrolytic treatment method	205/687	205/704		Uesugi, Akio	☑	☐	☐	☐	☐	☐	☐	US
7	☐	☐	US 6337136 B1	20020108	9	Aluminum alloy support for lithographic printing plate	428/472	101/459; 420/534;		Suzuki, Hideki et al.	☑	☐	☐	☐	☐	☐	☐	US
8	☐	☐	US 6316057 B1	20011113	11	Method for coating a surface	427/400	148/246; 148/272;		Hirayama, Martina et al.	☑	☐	☐	☐	☐	☐	☐	US
9	☐	☐	US 6309049 B1	20011030	32	Printing apparatus and method for imaging charged	347/55			Salmon, Peter C.	☑	☐	☐	☐	☐	☐	☐	US
10	☐	☐	US 6267870 B1	20010731	11	Treating aluminum workpieces	205/705	205/721		Amor, Martin Philip et al.	☑	☐	☐	☐	☐	☐	☐	US
11	☐	☐	US 6264765 B1	20010724	11	Method and apparatus for casting, hot rolling and	148/511	148/508; 148/574;		Bryant, J. Daniel et al.	☑	☐	☐	☐	☐	☐	☐	US

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combine with M1-M4 hydroxide

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65	US 5096790 A				☑					USPAT
66	US 5078805 A	6				☑				USPAT
67	US 5076899 A	11				☑				USPAT
68	US 5028276 A	10			☑					USPAT
69	US 5019188 A	3			☑					USPAT

thickness in a range from 10 to 50 mm, and is taken up by the coiler 4 so as to be formed into a coil. With respect to the conditions in the hot-rolling machine 3, the suitable temperature is in a range from 350.degree. to 550.degree. C. because the temperature gives an influence particularly on the electrolytic grain property of a support for a planographic printing plate .

Detailed Description Text - DETX (7):

Next, the thus obtained aluminum coil is cold-rolled so as to have a predetermined thickness. Steps of intermediate annealing, cold-rolling and the like may be further inserted in the producing process in accordance with the desired quality of the aluminum . Next, an aluminum support is formed from the aluminum coil through the steps of heat-treatment and correction, and then the obtained aluminum support is grained. The correction is sometimes included in the final cold-rolling step.

Detailed Description Text - DETX (8):

As the method of performing the graining on the support for a planographic printing plate according to the present invention, employed is a mechanical graining method, a chemical graining method, an electrochemical graining method, or any combination of the foregoing graining methods.

Detailed Description Text - DETX (11):

First of all, an aluminum support is etched by an alkaline. A preferable alkaline agent includes caustic soda, caustic potash, metasilicate soda, sodium carbonate, aluminate soda, gluconate soda or the like. It is preferable that a concentration of the alkaline agent is in the range from 0.01 to 20%, a temperature of the etching liquid is in the range from 20.degree. to 90.degree. C. and an etching period is in the range from 5 secs. to 5 mins. Also, a preferable etching amount is in the range from 0.01 to 5 g/m.sup.2, and regarding an aluminum support containing a relatively large amount of impurities, a preferable etching amount is in the range from 0.01 to 1 g/m.sup.2.

Detailed Description Text - DETX (12):

Additionally, if an insoluble smut remains on the surface of the aluminum plate, a desmut treatment may be performed, if necessary.

Detailed Description Text - DETX (13):

After pre-treatment as described above has been performed, AC electrolytic etching is performed to the aluminum plate in an electrolytic liquid mainly containing a hydrochloric acid or a nitric acid. Preferably, the frequency of the AC electrolytic current is selected to be in a range from 0.1 to 100 Hz, more preferably in a range from 0.1 to 1.0 Hz or from 10 to 60 Hz.

Detailed Description Text - DETX (14):

Preferably, the solution concentration is in a range from 3 to 150 g/l, more

a surface cutting step. Therefore, the cost of equipment decreases and the running cost also decreases.

Further, the support obtained according to the present invention has an excellent quality as a support for a planographic printing plate particularly using a photosensitive material.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic view for explaining a part of the process of the method of producing an aluminum support according to the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

An embodiment of the method of producing an aluminum support to be used according to the present invention will be described more specifically with reference to the schematic view of FIG. 1, which explains the producing process. An ingot is melted and held in a melting and holding furnace 1 so that the molten metal is sent to a casting machine 2 and hot-rolling machines 3. That is, a hot-rolled coil of a thin plate is directly formed from molten aluminum and taken up by a coiler 4.

The producing conditions in those parts will be described more in detail. It is necessary to maintain the temperature in the melting and holding furnace 1, i.e., the molten aluminum, to a value not lower than the melting point of aluminum. The melting point varies depending on the components of the aluminum alloy and generally takes a value of 800° C. or more.

Further, inclusions such as an oxide, etc., and alkali metals such as sodium, etc., are contained in the molten aluminum, and it is therefore necessary to remove such harmful materials. As the method of removing such harmful materials, flux treatment, chlorine treatment, etc., are generally used. As the flux, ethane hexachloride is most widely used.

Next, the molten aluminum is cast by the casting machine 2. There are various casting systems which are roughly grouped into a movable-mold system and a fixed-mold system. Almost all the current industrially-running casting methods are the Hunter method, the 3C method, the Hazley method, etc., which belong to the movable-mold system. Although the casting temperature is different between the movable-mold and fixed-mold methods from each other, the most suitable casting temperature is about 700° C. A 100-300 mm thick slab obtained in such a continuous casting method as described above is hot-rolled.

The hot-rolling machine 3 is constituted by breaking-down rolls and finishing rolls. The slab is hot-rolled so as to be formed into a strip having a thickness in a range from 10 to 50 mm, and is taken up by the coiler 4 so as to be formed into a coil. With respect to the conditions in the hot-rolling machine 3, the suitable temperature is in a range from 350° to 550° C. because the temperature gives an influence particularly on the electrolytic grain property of a support for a planographic printing plate.

Next, the thus obtained aluminum coil is cold-rolled so as to have a predetermined thickness. Steps of intermediate annealing, cold-rolling and the like may be further inserted in the producing process in accordance with the desired quality of the aluminum. Next, an aluminum support is formed from the aluminum coil through the steps of heat-treatment and correction, and then the obtained aluminum support is grained. The

correction is sometimes included in the final cold-rolling step.

As the method of performing the graining on the support for a planographic printing plate according to the present invention, employed is a mechanical graining method, a chemical graining method, an electrochemical graining method, or any combination of the foregoing graining methods.

As the mechanical graining method, known are, for example, ball graining, wire graining, brush graining, solution honing, etc. As the electrochemical graining method, an AC electrolytic etching method is generally used. As the current, a usual AC sinusoidal current or a special alternating current such as a square wave or the like is used. Further, etching treatment using a caustic soda or the like may be performed as the pretreatment for the electrochemical graining.

In performing electrochemical graining, it is preferable to perform graining by use of an AC current in an aqueous solution mainly containing a hydrochloric acid or a nitric acid. This electrochemical graining method will be described in detail hereunder.

First of all, an aluminum support is etched by an alkaline. A preferable alkaline agent includes caustic soda, caustic potash, metasilicate soda, sodium carbonate, aluminate soda, gluconate soda or the like. It is preferable that a concentration of the alkaline agent is in the range from 0.01 to 20%, a temperature of the etching liquid is in the range from 20° to 90° C. and an etching period is in the range from 5 secs. to 5 mins. Also, a preferable etching amount is in the range from 0.01 to 5 g/m², and regarding an aluminum support containing a relatively large amount of impurities, a preferable etching amount is in the range from 0.01 to 1 g/m².

Additionally, if an insoluble smut remains on the surface of the aluminum plate, a desmut treatment may be performed, if necessary.

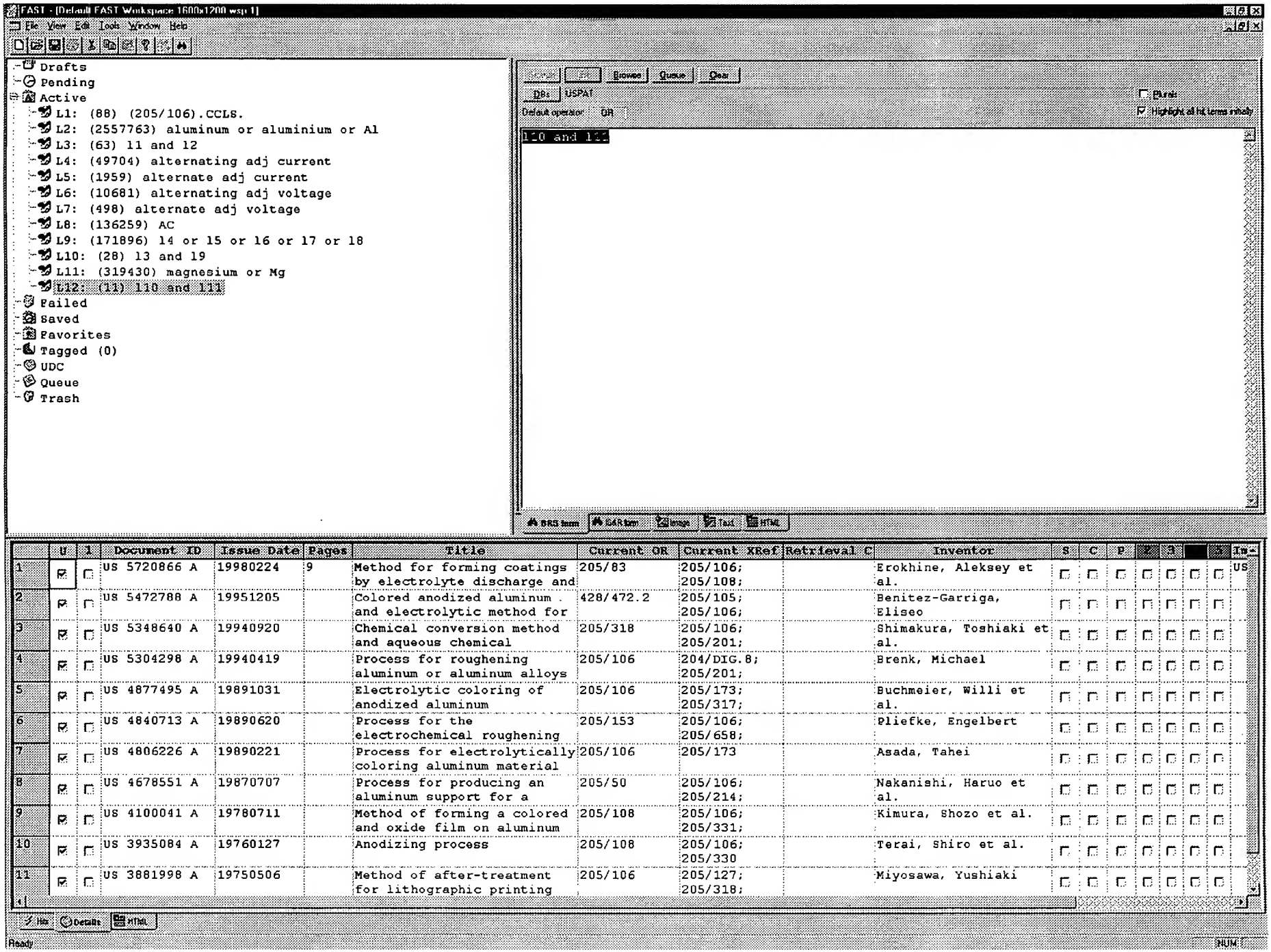
After pre-treatment as described above has been performed, AC electrolytic etching is performed to the aluminum plate in an electrolytic liquid mainly containing a hydrochloric acid or a nitric acid. Preferably, the frequency of the AC electrolytic current is selected to be in a range from 0.1 to 100 Hz, more preferably in a range from 0.1 to 1.0 Hz or from 10 to 60 Hz.

Preferably, the solution concentration is in a range from 3 to 150 g/l, more preferably in a range from 5 to 50 g/l. Preferably, the quantity of aluminum dissolution in the bath is not larger than 30 g/l, more preferably in a range from 2 to 20 g/l. An additive may be added if necessary. In the case of addition of an additive, however, it becomes difficult to control the solution concentration in mass production.

Preferably, the current density is selected to be in a range from 5 to 100 A/dm², more preferably in a range from 10 to 80 A/dm². Further, although the waveform of the power source may be properly selected in accordance with a desired quality and components of an aluminum support to be used, and so on. It is preferable to use such a special alternating waveform as disclosed in U.S. Pat. No. 4,087,344. The waveform and solution conditions are properly selected in accordance with the quantity of electricity, the desired quality, the compositions of an aluminum support to be used, and so on.

Next, the electrolytically grained aluminum is immersed in an alkali solution as a part of the desmutting treatment, thereby to dissolve smuts. As the alkali agent, there are various agents such as a caustic soda

Ex 1 anneal at 400°C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L5: (1959) alternate adj current
L6: (10681) alternating adj voltage
L7: (498) alternate adj voltage
L8: (136259) AC
L9: (171896) l4 or l5 or l6 or l7 or l8
L10: (28) l3 and l9
L11: (319430) magnesium or Mg
L12: (11) l10 and l11
Failed
Saved
Favorites
Tagged (0)
UDC
Queue
Trash
Search
Edit
Browse
Queue
Clear
DBs
USPAT
Default operator
OR
Plural
Highlight all hit terms initially
l10 and l11
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Text
HTML
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1
Document ID
Issue Date
Pages
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Current OR
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11 US 3881998 A 19750506 Method of after-treatment for lithographic printing 205/106 205/127; 205/318; Miyosawa, Yushiaki
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12 Al-Mg etch AC chemistry

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7	US 4806226 A	4				☑					USPAT

Detailed Description Text - DETX (12):

The aluminum substrates to which the aqueous chemical conversion solution of the present invention can be applied may be made of aluminum or its alloys such as an aluminum-copper alloy, an aluminum-zinc alloy, an aluminum-manganese alloy, an aluminum-magnesium alloy, an aluminum-magnesium-silicon alloy, an aluminum-zinc-magnesium alloy, etc. The chemical conversion solution can also be applied to metal members plated with aluminum.

Detailed Description Text - DETX (13):

The aluminum substrate may be in any shape such as a plate, a rod, a wire, a pipe, etc. Aluminum cans as well as aluminum caps of containers for food and beverages may also be treated with the aqueous chemical conversion solution of the present invention.

Detailed Description Text - DETX (16):

Before treating an aluminum substrate with acid or the alkali, a degreasing treatment is usually conducted on the aluminum substrate in the method of the present invention. The degreasing treatment may be conducted with a solvent such as trichloroethylene, perchloroethylene, gasoline, n-hexane, etc., or with an alkali solution of sodium hydroxide, sodium carbonate, sodium silicate, sodium phosphate, etc.

Detailed Description Text - DETX (17):

(B) Dissolution of Aluminum Oxide Layer

Detailed Description Text - DETX (18):

After degreasing, the aluminum substrate is rinsed with water and then treated with an acid or an alkali to dissolve an oxide layer thereof to increase the electrical conductivity of the aluminum substrate.

Detailed Description Text - DETX (19):

Specific examples of the acid usable in this treatment include phosphoric acid, sulfuric acid, nitric acid, etc. In view of the easiness of handling and the quality of the finished aluminum substrate, it is preferable to use phosphoric acid. As far as the dissolution of the oxide layer is concerned, better results are obtained in the order of hydrofluoric acid, hydrochloric acid, phosphoric acid, sulfuric acid and nitric acid (hydrofluoric acid: maximum). However, hydrofluoric acid and hydrochloric acid are not suitable for the method of the present invention, because the use of hydrofluoric acid causes fluoride ions to be introduced into the aqueous chemical conversion solution, and the use of a hydrochloric acid tends to generate pitting on the resulting film.

Detailed Description Text - DETX (20):

With respect to the concentration and temperature of the aqueous solution of

the predetermined minimum potential E_m at least once, in most cases, several times. Incidentally, the pulse width or the alternating frequency is not particularly limited. The alternating-potential variation patterns need not necessarily be in a triangular or rectangular pulse shape. They may also be in a shape of an exponentially decreasing curve, a sinusoidal curve, etc. as long as it reaches the predetermined minimum potential E_m at least once. A stepwise potential variation pattern is also applicable. Further, potential variation patterns obtained by combining two or more potential variation patterns shown in FIGS. 5 and 6 can also be used in the method of the present invention.

In the method of the present invention, a negative voltage V_f is applied to the aluminum substrate so that the potential of the aluminum substrate reaches the predetermined minimum potential E_m at least once. The shorter a period until the potential of the aluminum substrate reaches the predetermined minimum potential E_m during the immersion process, the more zinc phosphate crystal nuclei are deposited on the surface of the aluminum substrate, thereby forming a denser chemical conversion film. Accordingly, the application of voltage V_f is preferably conducted as immediately as possible after the immersion of the aluminum substrate in the aqueous chemical conversion solution to form a good chemical conversion film.

The immersion period during which the negative voltage V_f is applied to the aluminum substrate is preferably 15–300 seconds, more preferably 60–120 seconds.

After completing the chemical conversion treatment, the aluminum substrate is rinsed with water and dried at 90° C. for about 10 minutes.

A paint film can be coated on the resulting chemical conversion film of the aluminum substrate. Specific examples of the paint which can be applied onto the chemical conversion film include thermoset resin paints such as melamine alkyd resin paints, acrylic melamine resin paints, cationic electrodeposition paints such as epoxy resins, etc., and thermoplastic resin paints such as acrylic lacquer, etc.

The present invention will be explained in further detail by way of the following Examples without intention of restricting the scope of the claims.

In Examples, Comparative Examples and Reference examples, the following potential variation patterns and materials of the aluminum substrate were used.

Potential Variation Patterns

A voltage V_f was applied to each aluminum substrate in such a manner that the potential of the aluminum substrate changed in the following patterns:

(1) The potential dropped to a predetermined minimum potential E_m quickly after the immersion and was kept at the level of the predetermined minimum potential E_m as seen in FIG. 5 (*d*).

(2) The potential dropped to a predetermined minimum potential E_m quickly after the immersion, and then varied in a rectangular pulse manner between the predetermined minimum potential E_m and the natural electrode potential E_{Of} (pulse width: 10 seconds at both E_m and E_{Of}) as seen in FIG. 6 (*a*).

(3) The potential dropped to a predetermined minimum potential E_m quickly after the immersion, kept at the minimum potential E_m for about 30 seconds, and then returned to the natural electrode potential E_{Of} as seen in FIG. 5 (*f*).

(4) No voltage V_f was applied to the aluminum substrate so that the potential of the aluminum substrate remained at the natural electrode potential E_{Of} throughout the immersion process.

(5) The potential of the aluminum substrate was changed to 0.5 V higher than the natural electrode potential E_{Of} immediately after the immersion, kept at E_{Of} for about 30 seconds, and then fell to the natural electrode potential E_{Of}.

Predetermined Minimum Potential (E_m)

The predetermined minimum potential E_m of each aluminum substrate was set at a level which was lower than the initial natural electrode potential E_0 by 1.5 V, 1.3 V, 1.2 V, 1.1 V, 1.0 V, and 0.8 V, respectively, except for Comparative Example 3 in which the potential of the aluminum substrate was increased to 0.5 V higher than the initial natural electrode potential E_0 (reference electrode: Ag/AgCl).

Materials of Aluminum Substrate

Each aluminum substrate of 70 mm×10 mm×0.8 mm was made of the following material:

A: Aluminum Type 5000 (Al/Mg/Cu), or

B: Aluminum Type 6000 (Al/Mg/Cu/Si).

EXAMPLES 1-22, COMPARATIVE EXAMPLES 1-3, REFERENCE EXAMPLES 1 and 2

Each aluminum substrate was subjected to the following treatments:

(1) Degreasing

Alkali degreasing was conducted on each sample of the aluminum substrate with an alkali degreasing agent (Surfcleaner 53® available from Nippon Paint Co., Ltd.) at 45° C. for 2 minutes.

(2) Dissolution of Aluminum Oxide Layer

Each sample was then subjected to one of the following oxide layer-dissolution treatments:

(i) The sample was immersed in a phosphoric acid solution (concentration: 20 weight %) at 20° C. for 5 minutes.

(ii) The sample was immersed in a sulfuric acid solution (concentration: 5 weight %) at 20° C. for 10 minutes.

(iii) The sample was immersed in an aqueous solution of sodium hydroxide (concentration: 5 weight %) at 20° C. for 5 minutes.

(3) Rinsing

Each sample was then rinsed with a tap water at room temperature for about 15 seconds.

(4) Surface Conditioning Treatment

After the rinsing, each sample was subjected to a surface conditioning treatment with Surfline 5N-10® available from Nippon Paint Co., Ltd.) at 20° C. for 20 seconds.

(5) Chemical Conversion Treatment

The following aqueous phosphate-based chemical conversion solutions were prepared for treating each sample.

Solution 1

An aqueous, phosphate-based chemical conversion solution substantially free from fluoride ions and con-

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color Al with AC in soln having pH of 5-9

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	Document ID	Pages	1	2	3	4	5	6	S	C	P	Kind Codes	Sou
2	US 5472788 A	18							☑				USPAT
3	US 5348640 A	13							☑				USPAT
4	US 5304298 A	10							☑				USPAT
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6	US 4840713 A	7							☑				USPAT
	US 4806226 A	8							☑				USPAT
8	US 4678551 A	11							☑				USPAT

US-PAT-NO: 4806226

DOCUMENT-IDENTIFIER: US 4806226 A

TITLE: Process for electrolytically coloring aluminum material

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TITLE - TI (1):

Process for electrolytically coloring aluminum material

Abstract Text - ABTX (1):

A process for electrolytically coloring an aluminum material wherein a base aluminum or aluminum alloy is anodized and the anodized material is electrolyzed in a second electrolytic coloring step using a bath containing nickel and zinc salts, a chelating reagent for nickel ions and a supporting electrolyte at a pH of 4.5 or greater using alternating, direct or dual alternating-direct current electrolytic processes. A molybdate may also be employed in the secondary electrolysis process and the resultant coating has a color ranging from a grey series to black.

Brief Summary Text - BSTX (3):

This invention relates to a process for producing a corrosion resistant colored surface on an aluminum or aluminum alloy substrate.

Brief Summary Text - BSTX (5):

Anodized aluminum, formed by the electrolytic treatment of aluminum or aluminum alloys in a sulfuric acid bath may be treated by a secondry electrolytic treatment in a coloring bath containing metallic salts, as is described in U.S. Pat. No. 3,382,160. It is believed that this process results in the precipitation of the metal salts in the pores of the anodic coating on the aluminum substrate. Aluminum treated by this process produces colored materials useful for construction and other applications, but the color series which may be obtained is limited to a bronze series merging into black.

Brief Summary Text - BSTX (6):

The codeposition of nickel and zinc from a plating bath containing nickel sulfate and zinc sulfate, at a pH of 2-4, onto has been reported by K. Mitsuo et al, Electrochemistry 45, No. 12 (1977), pp. 728-733 and Electrochemistry 47, No. 2 (1979) pp. 89-94. According to these papers, the plated coating of an intermetallic compound of nickel and zinc are formed on a rolled thin copper plate. The color of the plated coating disclosed therein has a silver-white tint and it has not been found possible heretofore, using such a plating process, to obtain a pure grey series coating.

Brief Summary Text - BSTX (7):

PROCESS FOR ELECTROLYTICALLY COLORING ALUMINUM MATERIAL

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a process for producing a corrosion resistant colored surface on an aluminum or aluminum alloy substrate.

2. Description of the Prior Art

Anodized aluminum, formed by the electrolytic treatment of aluminum or aluminum alloys in a sulfuric acid bath may be treated by a secondry electrolytic treatment in a coloring bath containing metallic salts, as is described in U.S. Pat. No. 3,382,160. It is believed that this process results in the precipitation of the metal salts in the pores of the anodic coating on the aluminum substrate. Aluminum treated by this process produces colored materials useful for construction and other applications, but the color series which may be obtained is limited to a bronze series merging into black.

The codeposition of nickel and zinc from a plating bath containing nickel sulfate and zinc sulfate, at a pH of 2-4, onto has been reported by K. Mitsuo et al, *Electrochemistry* 45, No. 12 (1977), pp. 728-733 and *Electrochemistry* 47, No. 2 (1979) pp. 89-94. According to these papers, the plated coating of an intermetallic compound of nickel and zinc are formed on a rolled thin copper plate. The color of the plated coating disclosed therein has a silver-white tint and it has not been found possible heretofore, using such a plating process, to obtain a pure grey series coating.

There remains a long-felt need to prepare secondarily colored anodized aluminum products of a true grey series, without bronze tint and with enhanced corrosion resistance.

SUMMARY OF THE INVENTION

Applicants have discovered that it is possible to prepare a grey series secondary electrolytic coating on anodized aluminum by preparing an electrolytic coloring bath containing mixtures of nickel and zinc salts as a coloring agent, by the addition thereto of a chelating agent for nickel ions, by adjustment of the pH of the bath by selection of a suitable supporting electrolyte in the bath and by the application of an electric current. The color coated aluminum materials according to this invention provide a grey series to black coating which also has superior corrosion resistance to the secondary electrolytically coated products of the prior art.

According to applicant's process, aluminum or an aluminum alloy (hereinafter "aluminum material") is first anodized to form an anodized aluminum material and the anodized aluminum material is treated secondarily, using alternating or direct currents, or dual currents of both alternating and direct current, in a coloring bath having a pH of 4.5 or greater, containing the coloring agent, chelating agent and supporting electrolyte. The coloring agent may be further modified by the addition of a molybdate salt.

A "chelating agent" for nickel ions may be any chemical compound that acts to stabilize nickel ions so that the rate of deposition of nickel ions during the secondary electrolytic coloring process is controlled to effect the codeposition of zinc and nickel during the treating process in ratios of nickel to zinc which produce a grey color.

DETAILED DESCRIPTION OF THE INVENTION

The anodic treatment of aluminum materials in electrolytes, typically acids, produces an oxide film of substantial thickness and abrasion resistance. The oxide coating is integral with the aluminum and adheres tightly to the base substrate. The coating is an amorphous material having minute pores. Secondary treatment of the coating is usually an electrolytically coloring process.

When the secondary treatment is the electrolytic coloring process, such as that described in U.S. Pat. No. 3,382,160 reference, a coating of superior corrosion resistance is obtained but can be varied in the darkness of the coating but which contains an unavoidable bronze tint to the point where the color merges to black.

According to the process of this invention, a secondary electrolytic treatment is performed using an electrolytic coloring bath containing a combination of nickel and zinc salts, preferably in a specific weight ratio based upon the content of nickel and zinc, a chelating reagent for nickel ions and a suitable supporting electrolyte.

The coloring agent which is used contains nickel salts, preferably nickel sulfate, ammonium nickel sulfate or nickel sulfamate. Zinc salts, preferably zinc sulfate or zinc chloride are also required in the coloring bath.

The chelating reagent for nickel ions is any chemical compound which acts to stabilize nickel ions so that the rate of deposition of the nickel ions may be controlled during the deposition of zinc ions. Preferred chelating agents are gluconic acid, maleonic acid, sulfosalicylic acid, tartaric acid, citric acid, sulfophthalic acid, sulfamic acid, and boric acid.

A supporting electrolyte is also required in the bath. Preferred supporting electrolytes are ammonium sulfate, magnesium sulfate and other sulfates which do not affect the nickel to zinc ratio.

The pH of the bath is at least 4.5 and is preferably within the range of 5 to 9.

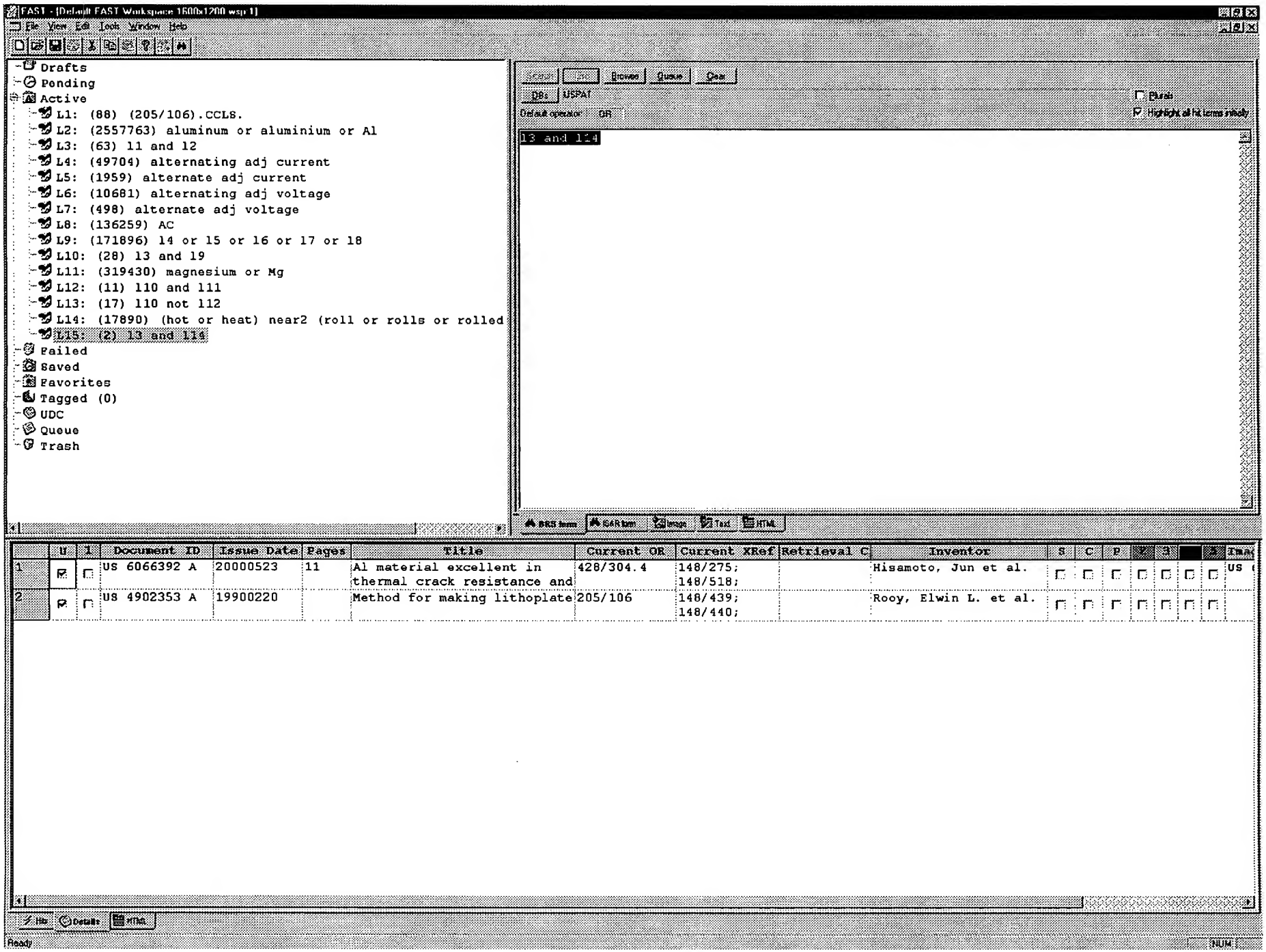
In addition to the above, it has been found that the addition of a molybdate salt, such as ammonium molybdate, is advantageous in producing a grey series coloration.

In the secondary electrolytic treatment, a direct current or an alternating current may be used and it is also possible to use dual currents of both alternating and direct current. Voltage differences of 10 to 30 volts are preferred at a bath temperature of 15° to 30° C. The anodized aluminum material is used as one electrode during the secondary electrolytic treatment. The other electrode may be any electrode which does not produce contaminating ions, and is preferably nickel or carbon.

Although not wishing to be bound by any particular theory, applicant hypothesizes that the following principles serve to explain the operation of applicant's invention.

Aluminum has a strong negative polarity in electrolytic treatment. As a result, the positive ions of metals in the treatment bath are strongly attracted to the aluminum and form a concentration gradient throughout the bath, analogous to the conditions observed during plating operations.

Mitsuo et al. reported, in the articles cited above, that codeposition of nickel and zinc occurred during plating from a bath containing nickel sulfate and zinc sulfate at a pH between 2 and 4, and they hypothesized that inter-



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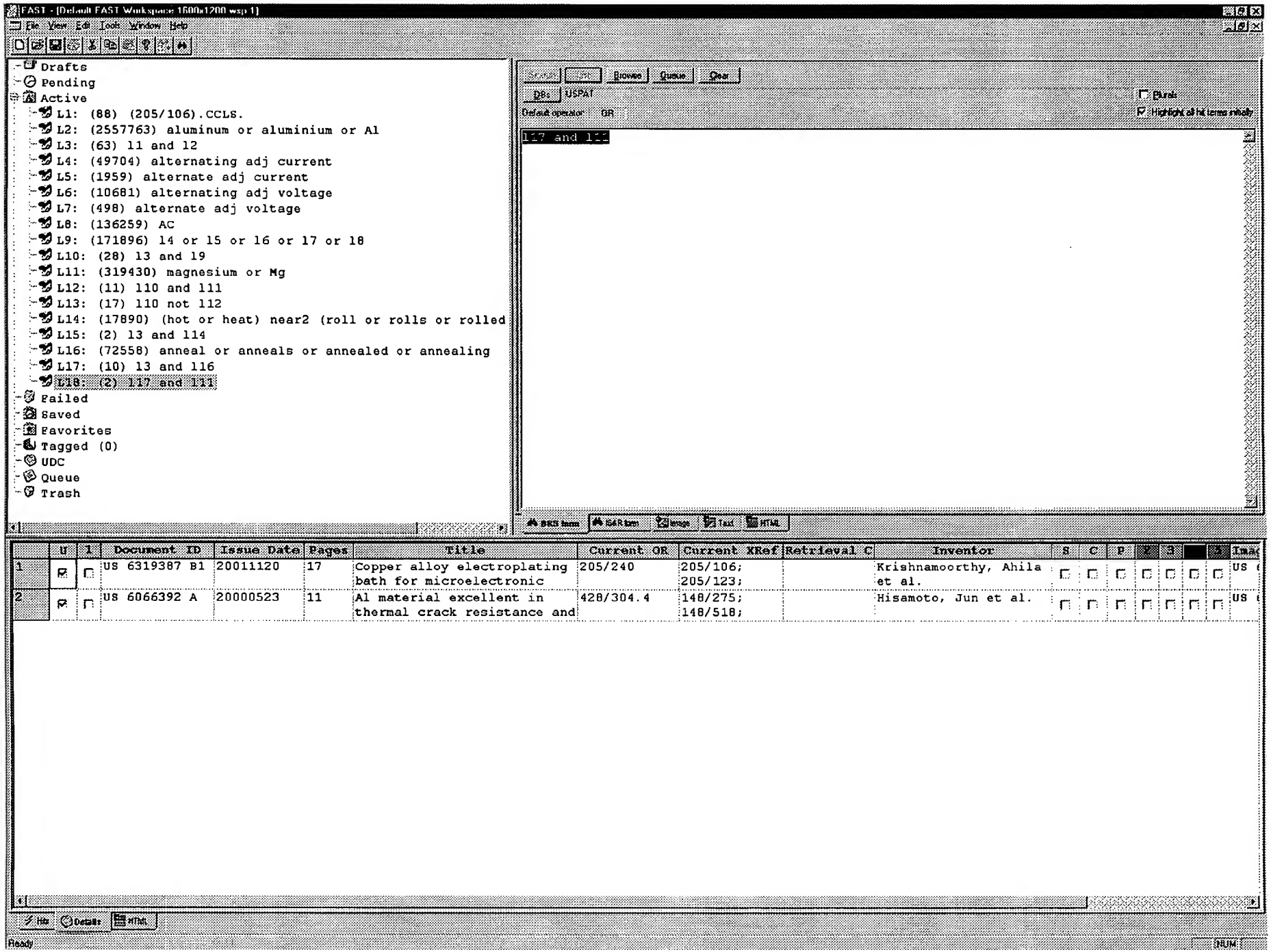
2	☑	☐	US 4902353 A	19900220		Method for making lithoplate	205/106	148/439; 148/440;		Rooy, Elwin L. et al.	☐	☐	☐	☐	☐	☐	☐	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L5: (1959) alternate adj current
L6: (10681) alternating adj voltage
L7: (498) alternate adj voltage
L8: (136259) AC
L9: (171896) l4 or l5 or l6 or l7 or l8
L10: (28) l3 and l9
L11: (319430) magnesium or Mg
L12: (11) l10 and l11
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L14: (17890) (hot or heat) near2 (roll or rolls or rolled
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L16: (72558) anneal or anneals or annealed or annealing
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Copper alloy electroplating bath for microelectronic
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Krishnamoorthy, Ahila et al.
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Al material excellent in thermal crack resistance and
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Hisamoto, Jun et al.
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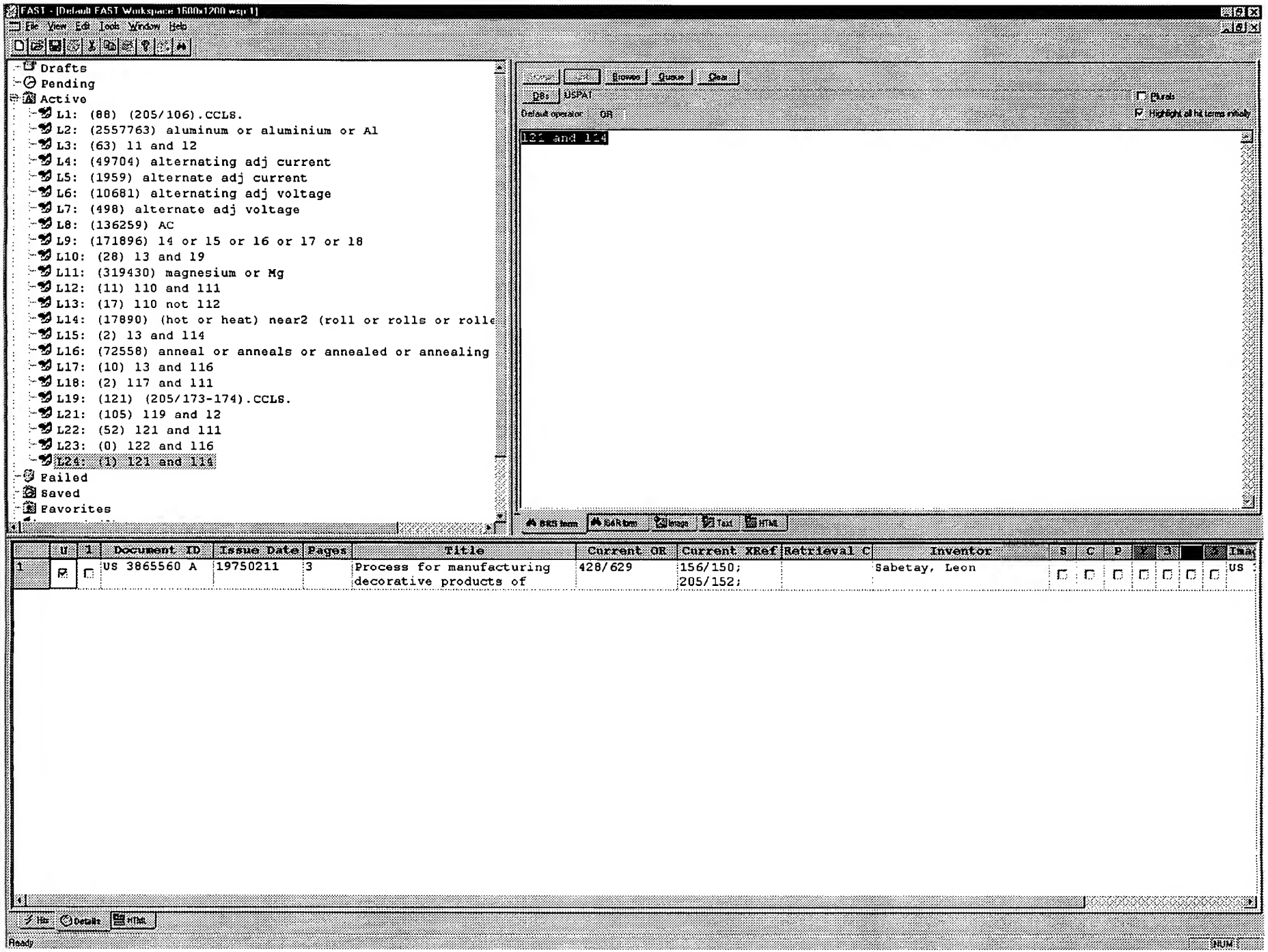
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L3: (63) l1 and l2
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L16: (72558) anneal or anneals or annealed or annealing
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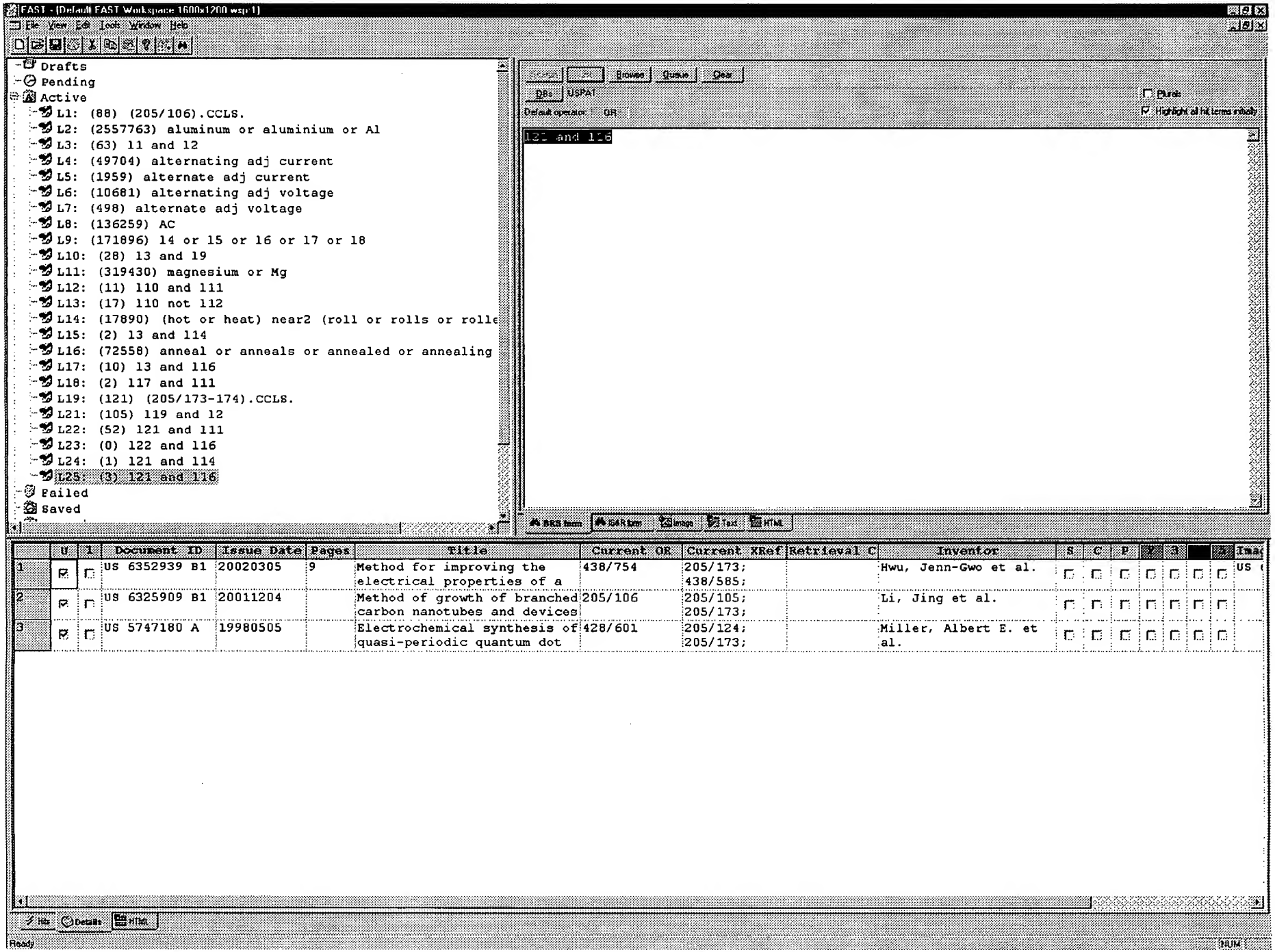

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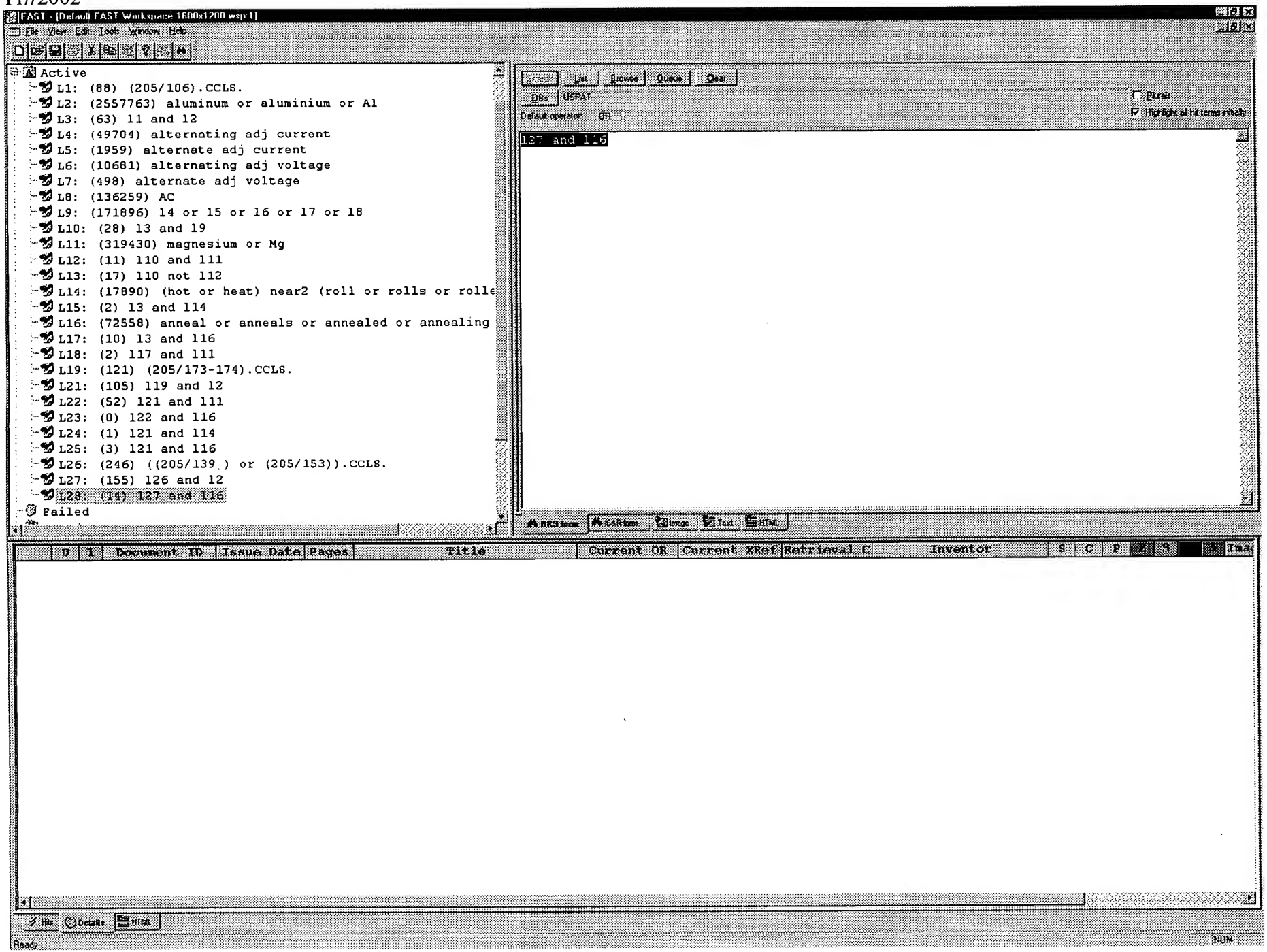
1	☑	☐	US 6352939 B1	20020305	9	Method for improving the electrical properties of a	438/754	205/173; 438/585;		Hwu, Jenn-Gwo et al.	☐	☐	☐	☐	☐	☐	☐	US
2	☑	☐	US 6325909 B1	20011204		Method of growth of branched carbon nanotubes and devices	205/106	205/105; 205/173;		Li, Jing et al.	☐	☐	☐	☐	☐	☐	☐	
3	☑	☐	US 5747180 A	19980505		Electrochemical synthesis of quasi-periodic quantum dot	428/601	205/124; 205/173;		Miller, Albert E. et al.	☐	☐	☐	☐	☐	☐	☐	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L2: (2557763) aluminum or aluminium or Al
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L19: (121) (205/173-174).CCLS.
L21: (105) l19 and l2
L22: (52) l21 and l11
L23: (0) l22 and l16
L24: (1) l21 and l14
L25: (3) l21 and l16
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7	US 4470885 A	3							☑				USPAT
8	US 4426260 A	4							☑				USPAT
9	US 4276127 A	8							☑				USPAT

treatment, there is a significant improvement in the leakage current characteristics of the resultant electrolytic capacitor. It has further been found that by exposing the etched aluminum foil to 5 to 40% nitric acid at a temperature of about 25.degree. C. to 95.degree. C. for a period of about 10 seconds to 30 minutes the leakage current characteristics of the capacitor employing such a foil is at least as good as that produced by the method of the Japanese patent application.

Brief Summary Text - BSTX (18):

While improved results may be obtained with aluminum foil of an iron content of greater than 0.010% by weight it has been found that the greatest improvements have been found where the iron content is greater than about 0.025% and the aluminum content is 98%-99.95% by weight.

Brief Summary Text - BSTX (19):

In order to improve the mechanical properties thereof it is preferable to anneal the foil subsequent to the treatment with the nitric acid and after rinsing and drying by heating the aluminum foil at 250.degree. C. to 600.degree. C. for 5 minutes to 12 hours.

Brief Summary Text - BSTX (20):

However, it should be noted that the annealing treatment does not significantly increase the leakage current or the capacitance characteristics of the capacitor.

Detailed Description Text - DETX (4):

A section of a 9 micron thick 1193 aluminum foil, having a aluminum content of 99.93% by weight and an iron content of three hundred parts per million, is electrolytically etched according to standard procedures in an aqueous salt solution. The etched aluminum foil is then washed with ion-free water and dried at 85.degree. to 100.degree. C. The aluminum foil is then immersed for about 1 to 3 minutes specifically for 1 minute in a 20% solution of nitric acid maintained at a temperature of 65.degree. C. followed by a deionized water rinse and a drying step.

Detailed Description Text - DETX (6):

The foil is then annealed by heating at 450.degree. C. for five minutes.

Detailed Description Text - DETX (8):

This value may be compared to a sample of etched 99.96% aluminum which had a leakage of 0.75 .mu.A/Vuf after identical treatment.

Detailed Description Text - DETX (10):

A section of an 1193 aluminum foil similar to that employed in Example I is

United States Patent [19]

Stevens

[11] **4,426,260**

[45] **Jan. 17, 1984**

[54] **PREPARATION OF ALUMINUM ELECTROLYTIC CAPACITOR FOIL**

[75] Inventor: James L. Stevens, Irmo, S.C.

[73] Assignee: U.S. Philips Corporation, New York, N.Y.

[21] Appl. No.: 405,668

[22] Filed: Aug. 6, 1982

[51] Int. Cl.³ C25D 5/44; C25D 5/50; C25D 11/08

[52] U.S. Cl. ..... 204/33; 204/37 R; 204/58

[58] Field of Search ..... 204/33, 38 A, 58, 37 R; 361/433

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Primary Examiner—G. L. Kaplan
Assistant Examiner—Nam X. Nguyen
Attorney, Agent, or Firm—Norman N. Spain

[57] **ABSTRACT**

The invention involves a method of preparing aluminum foil for use as an anode in electrolytic capacitors. The resultant electrolytic capacitor is improved by subjecting the etched aluminum foil to a heat treatment of 100° to 600° C. for about 10 seconds to 20 minutes prior to the treatment with nitric acid.

7 Claims, No Drawings

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6	US 4482434 A	7						☑				USPAT
7	US 4470885 A	3						☑				USPAT
8	US 4426260 A	4						☑				USPAT
9	US 4276127 A	8						☑				USPAT
10	US 4248674 A	5						☑				USPAT
11	US 4183788 A	6						☑				USPAT
12	US 4017265 A	12						☑				USPAT

pairs of tandem brushes with an aqueous slurry of unfused crystalline alumina fed from recirculating sumps. Suitable graining equipment is commercially available from the Fuller Brush Company and was used in the examples described herein.

Brief Summary Text - BSTX (23):

Anodizing following the graining operation of the invention may be carried out using known techniques to form a porous anodic oxide layer on the grained aluminum surface. Sulfuric acid is the preferred electrolyte. See Kirk-Othmer Encyclopedia of Chemical Technology, 2nd Ed., Vol. 1, p. 978 et seq.

Brief Summary Text - BSTX (24):

Cold rolled aluminum should be employed for forming printing plates according to the invention. Softer aluminum is not suitable because it will tear or rip when engaged by the lock-up device of a printing press. Preferred aluminum sheet generally has a temper of between H12 and H19 where direct cold reduction is employed or between H22 and H27 where a combination of cold reduction and back (annealing) are employed, as specified by the American Aluminum Association in Aluminum Standards and Data, published by the Association.

Brief Summary Text - BSTX (25):

Aluminum printing plates can be made in any fashion known in the art, for example as taught by the following

Brief Summary Text - BSTX (26):

U.S. Pat. No. 2,714,066, Jewitt et al, July 26, 1955;

Brief Summary Text - BSTX (34):

Especially preferred is an anodically oxidized aluminum base having an aluminum oxide surface which is initially porous after anodic oxidation and subsequently treated with an alkali metal silicate and sealed prior to application of a light-sensitive coating. This is the subject of U.S. Pat. No. 3,181,461 referred to above.

Brief Summary Text - BSTX (35):

It is preferred to continuously anodize aluminum after graining utilizing the anodizing techniques described in patents U.S. Pat. No. 3,865,700 issued Feb. 11, 1975, and U.S. Pat. No. 3,920,525 issued Nov. 18, 1975. If desired, the aluminum base can be provided with a composite anodized and discontinuously electroplated surface prior to application of the light-sensitive coating as taught in patent U.S. Pat. No. 3,929,594 issued Dec. 30, 1975.

Detailed Description Text - DETX (3):

Multiple graining units are installed in a continuous web anodizing line. The placement of these units relative to the entire line is after the degreasing

United States Patent [19]

Fromson et al.

[11] **4,183,788**

[45] **Jan. 15, 1980**

[54] **PROCESS FOR GRAINING AN ALUMINUM BASE LITHOGRAPHIC PLATE AND ARTICLE THEREOF**

[75] Inventors: **Howard A. Fromson, 15 Rogues Ridge Rd., Weston, Conn. 06880; Robert F. Gracia, Scituate, Mass.**

[73] Assignee: **Howard A. Fromson, Weston, Conn.**

[21] Appl. No.: **881,991**

[22] Filed: **Feb. 28, 1978**

[51] Int. Cl.[2] **B41N 3/04; G03C 1/94**

[52] U.S. Cl. ..... **430/278; 204/38 A; 204/29; 101/454; 101/455; 101/456; 101/459; 101/463**

[58] Field of Search ..... **204/28, 33, 38 A, 29; 51/309; 101/454-457, 459, 463; 96/86 R, 86 P**

[56] **References Cited**

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3,121,623	2/1964	Nesin	51/309
3,691,030	9/1972	Strossynski	204/32 R
3,834,998	10/1974	Watanabe	204/33
3,902,976	9/1975	Walls	204/38 A

Primary Examiner—John H. Mack
Assistant Examiner—William Leader
Attorney, Agent, or Firm—Sprung, Felfe, Horn, Lynch & Kramer

[57] **ABSTRACT**

Planar surfaces are roughened by graining with an aqueous slurry of unfused platy, crystalline alumina. Preferably, an aluminum base with is adapted to receive a light-sensitive coating thereon to make lithographic plate is grained with said aqueous slurry.

14 Claims, No Drawings

Al-4%Mn anneal

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8	US 4426260 A	4							☑				USPAT
9	US 4276127 A	8							☑				USPAT
10	US 4248674 A	5							☑				USPAT
11	US 4183788 A	6							☑				USPAT
12	US 4017265 A	12							☑				USPAT
13	US 3773579 A	4							☑				USPAT
14	US 3676309 A	7							☑				USPAT

US-PAT-NO: 4017265

DOCUMENT-IDENTIFIER: US 4017265 A

TITLE: Ferromagnetic memory layer, methods of making and adhering it to substrates, magnetic tapes, and other products

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Brief Summary Text - BSTX (46):

A disc about 14.5" in diameter having a central mounting operative of about 6.1" in diameter and a thickness of about 0.06" made of aluminum or an alloy of aluminum containing 4.0% magnesium and 0.5% manganese is sanded and machined to insure concentricity. The disc is then annealed and surface finished with diamond tools to produce an overall flatness of 0.002-0.003 inch and a surface finish of 1.0-1.5 microninches, arithmetic average.

Detailed Description Text - DETX (50):

An activator or reductant solution is prepared by dissolving 10 g anhydrous stannous chloride into 10 ml dimethylformamide. 0.10 g of this activator solution is diluted wth 20 ml 1,4 dioxane and two drops cyclohexanone and the diluted activator is coated with a 140 Q gravure roll onto a carbon pigmented Saran layer on polyester film. The carbon pigmented layer was made as follows by loading a one gallon stainless steel cannister with 8 lb nickel shot, 100 g carbon (Columbian Carbon Peerless 155 beads), 500 ml dimethylformamide and 15 g aluminum resinate (Meyers). The cannister is agitated for two hours. The dispersed carbon is loaded with resin by adding to the cannister during agitation, the resin comprising 200 g Saran F130, 800 ml methylethylketone, 350 ml cyclohexanone and 350 ml ethyl acetate.

Current US Cross Reference Classification - CCXR (2):

205/139

US Reference Patentee Name - URNM (2):

Schneble et al.

US Reference Patentee Name - URNM (3):

Koretzky et al.

US Reference Patentee Name - URNM (6):

Kovac et al.

US Reference Patentee Name - URNM (8):

United States Patent [19]

Taylor

[11] **4,017,265**

[45] **Apr. 12, 1977**

[54] **FERROMAGNETIC MEMORY LAYER, METHODS OF MAKING AND ADHERING IT TO SUBSTRATES, MAGNETIC TAPES, AND OTHER PRODUCTS**

[76] Inventor: **David W. Taylor, P.O. Box 67, Providence Road, Edgemont, Pa. 19028**

[22] Filed: **Feb. 15, 1972**

[21] Appl. No.: **226,512**

[52] U.S. Cl. ..... 428/675; 204/20; 204/30; 204/28; 204/29; 204/38 B; 204/38 E; 204/48; 340/174 TP; 427/304; 427/305; 427/306; 427/132; 428/928

[51] Int. Cl.² C25D 5/56; C25D 5/00; C25D 5/10; C25D 5/24

[58] Field of Search ..... 204/38 B, 38 E, 29, 204/30, 48, 20; 117/47 R, 130 E, 71, 227, 218, 235-240; 29/195 R, 199, DIG. 12, 203 MM; 106/1; 274/41.4; 340/174 TF

[56] **References Cited**

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3,227,635	1/1966	Koretzky et al.	204/48
3,269,854	8/1966	Hei	274/41.4
3,483,725	12/1969	Koretzky	204/48
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3,523,824	8/1970	Powers et al.	117/217
3,661,783	5/1972	Cooper	117/47 R

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"Electroplating of Co & Co Alloys–A Review," by Morral, Plating, 6–67, pp. 693, 696.

"Modern Electroplating," by F. A. Lowenheinn, 1963, pp. 143–144.

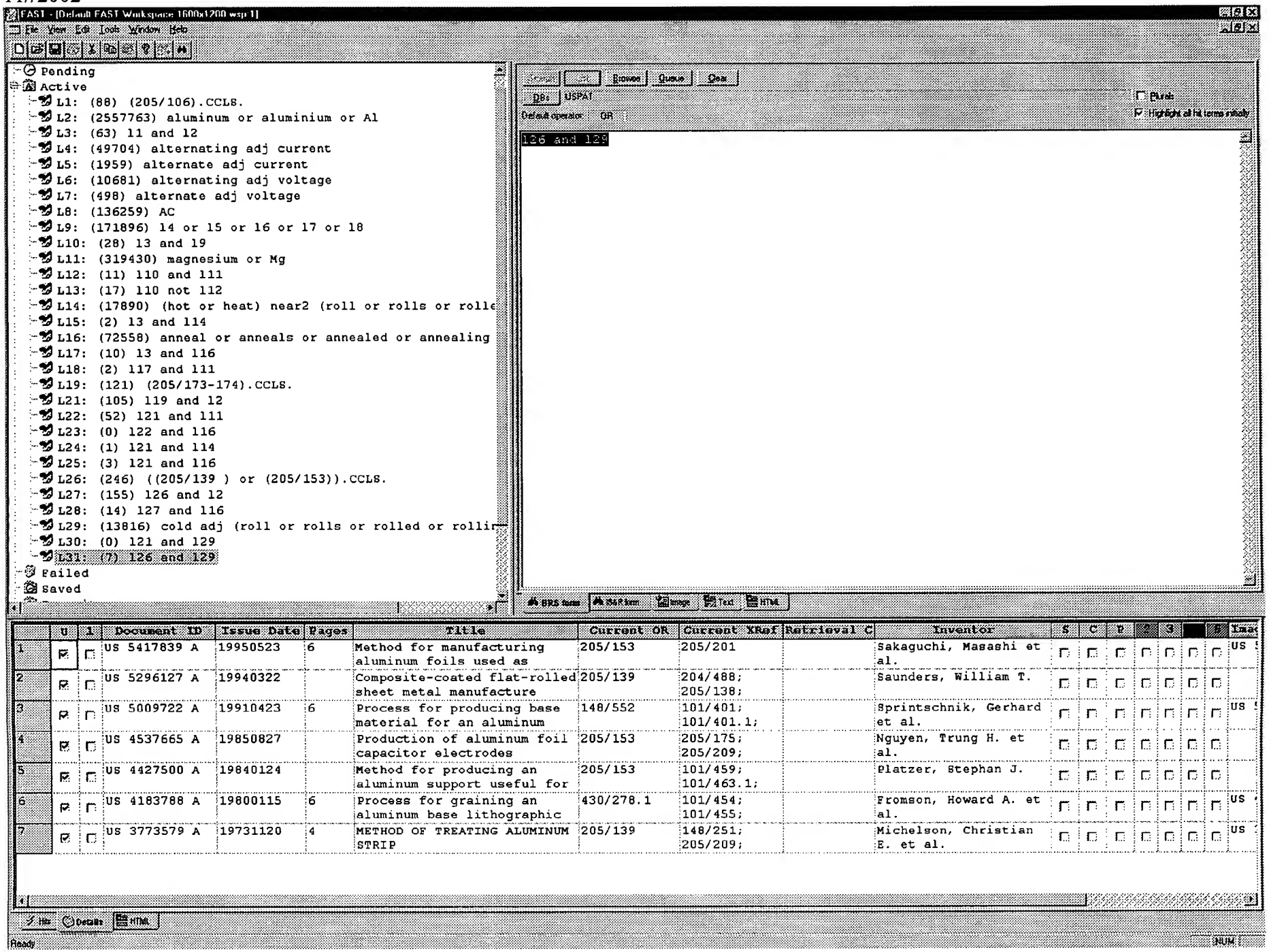
Primary Examiner—R. L. Andrews

Attorney, Agent, or Firm—Weiser, Stapler & Spivak

[57] **ABSTRACT**

A thin, ferromagnetic layer which can be continuous, substantially pit-free and uniform, said layer having high bit density capabilities consisting essentially of cobalt in the form of close packed hexagonal crystals. A substrate is treated with a catalytic activator upon which is deposited an electroless conductive layer, such as copper; the cobalt layer is deposited by electroplating on the conductive layer. The ferromagnetic layer has a nominal coercivity of about 200 to about 500 oersteds and exhibits no anisotropy in the plane of deposition.

40 Claims, No Drawings



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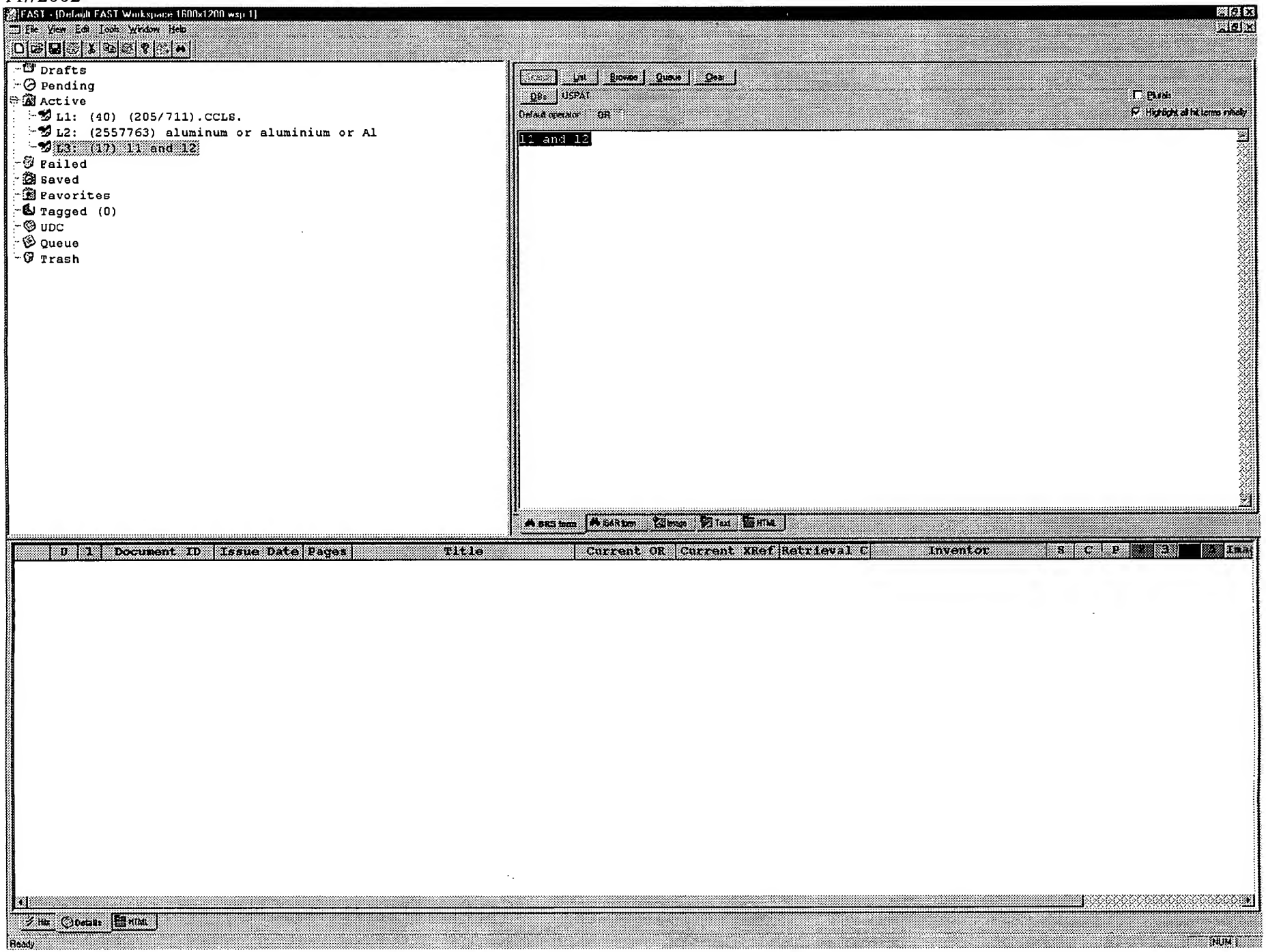
1	☑	☐	US 5417839 A	19950523	6	Method for manufacturing aluminum foils used as	205/153	205/201		Sakaguchi, Masashi et al.	☐	☐	☐	☐	☐	☐	☐	US
2	☑	☐	US 5296127 A	19940322		Composite-coated flat-rolled sheet metal manufacture	205/139	204/488; 205/138;		Saunders, William T.	☐	☐	☐	☐	☐	☐	☐	
3	☑	☐	US 5009722 A	19910423	6	Process for producing base material for an aluminum	148/552	101/401; 101/401.1;		Sprintschnik, Gerhard et al.	☐	☐	☐	☐	☐	☐	☐	US
4	☑	☐	US 4537665 A	19850827		Production of aluminum foil capacitor electrodes	205/153	205/175; 205/209;		Nguyen, Trung H. et al.	☐	☐	☐	☐	☐	☐	☐	
5	☑	☐	US 4427500 A	19840124		Method for producing an aluminum support useful for	205/153	101/459; 101/463.1;		Platzer, Stephan J.	☐	☐	☐	☐	☐	☐	☐	
6	☑	☐	US 4183788 A	19800115	6	Process for graining an aluminum base lithographic	430/278.1	101/454; 101/455;		Fromson, Howard A. et al.	☐	☐	☐	☐	☐	☐	☐	US
7	☑	☐	US 3773579 A	19731120	4	METHOD OF TREATING ALUMINUM STRIP	205/139	148/251; 205/209;		Michelson, Christian E. et al.	☐	☐	☐	☐	☐	☐	☐	US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L3: (17) l1 and l2
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13	US 4919774 A	9							☑					USPAT
14	US 4713153 A	11							☑					USPAT
15	US 4152471 A	22							☑					USPAT
16	US 4082868 A	18							☑					USPAT
17	US 4081344 A	11							☑					USPAT

the bath temperature, pH, concentration and the voltage used are the same as those used in the above, but the electrodes used are made of Ta, Nb, Zr, Al, Ti, or their alloys over which an oxidized film of 0.2 .mu. or more is provided by anodic oxidization treatment and the arrangement of the electrodes and their ratio are controlled such that the ratio of the coating deposit current of normal direction (plus portion to that of reverse direction (minus portion) on the inlet side of the electrodeposition bath is between 1:0.1 and 1:1 and such that the minus current portion is as a whole, one tenth or less of the plus current portion. Under these conditions, the pretreatment for electrodeposition coating and the subsequent alternating current electrodeposition coating are effected.

Drawing Description Text - DRTX (6):

FIG. 5 shows a relation between the bath voltage E.sub.2 and the bath current I.sub.2 when the pretreatment for electrodeposition coating is effected by the use of an electrode of an oxidized film type which has a condenser effect enlarged by an oxidized film of 0.2 .mu. or more produced by anodic oxidation of Ta, Nb, Zr, Al, Ti or their alloy.

Detailed Description Text - DETX (1):

In FIG. 3 showing an example of repair coating of the easy-open end, hereinafter referred to as the can end, according to this invention, the numeral 10 is an electrolytic cell, 6 is a guide for moving the can end which is concurrently utilized for passing electric current, 7 is a unit for moving the can end, 9 is an electrode having an oxidized film and 8 is the can end. The can ends 8 are transferred in the direction of arrows shown, while being subjected to (a) the pretreatment using A.C. electrodeposition coating followed by the A.C. electrodeposition coating, or (b) the pretreatment using A.C. electrodeposition coating followed by the D.C. electrodeposition coating, or (c) the pretreatment using A.C. electrodeposition coating followed by the A.C. electrodeposition coating by the use of an electrode having an oxidized film of 0.2 .mu. or more obtained by anodic oxidation of Ta, Nb, Zr, Al, Ti or their alloy.

Detailed Description Text - DETX (11):

As for the adjustment of the pH of the bath, if the extreme change in the electrodeposition property due to the melt-out of the base metal into the bath is not prevented, a perfect electrodeposition repair coating to be done in such short time as 15 seconds can not be practised for the can ends which are conveyed continuously into the bath. In this case, if the base metal is aluminium, tin, or tin-free steel, the pH should be adjusted to a range of 6 to 10. The concentration of the coating should preferably be thin in case of such a complicate shape as the can end from the viewpoint of the washability or of the saving of the coating. The velocity of deposition of the coating tends to be lowered if the concentration is less than 2%. Accordingly, it should be maintained at 2 to 20%.

Detailed Description Text - DETX (23):

In the second-mentioned aspect of the invention, the pretreatment for electrodeposition coating is conducted on the inlet side of the alternating

United States Patent [19]

Shindou et al.

[11] **4,081,344**

[45] **Mar. 28, 1978**

[54] **METHOD FOR ELECTRODEPOSITION REPAIR COATING OF THE END OF AN EASY-OPEN CAN**

[75] Inventors: Yoshio Shindou; Makoto Nakazura, both of Yokohama, Japan

[73] Assignee: Nippon Steel Corporation, Tokyo, Japan

[21] Appl. No.: 649,977

[22] Filed: Jan. 19, 1976

[30] Foreign Application Priority Data

Jan. 20, 1975 Japan 50-7847

[51] Int. Cl.² C25D 13/18; C25D 13/20

[52] U.S. Cl. ..... 204/181 R

[58] Field of Search 204/181

[56] References Cited

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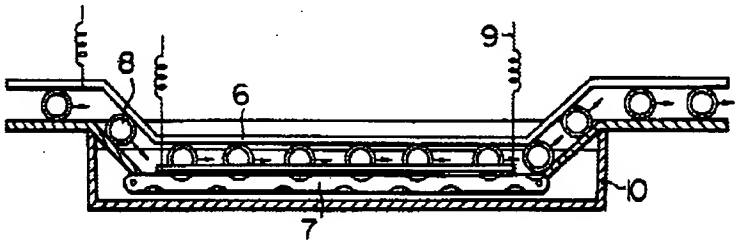
3,622,485	11/1971	Preece et al.	204/181
3,647,786	11/1974	Landauer et al.	204/181
3,878,078	4/1975	Tanaka et al.	204/181
4,005,000	1/1977	Kratka	204/181

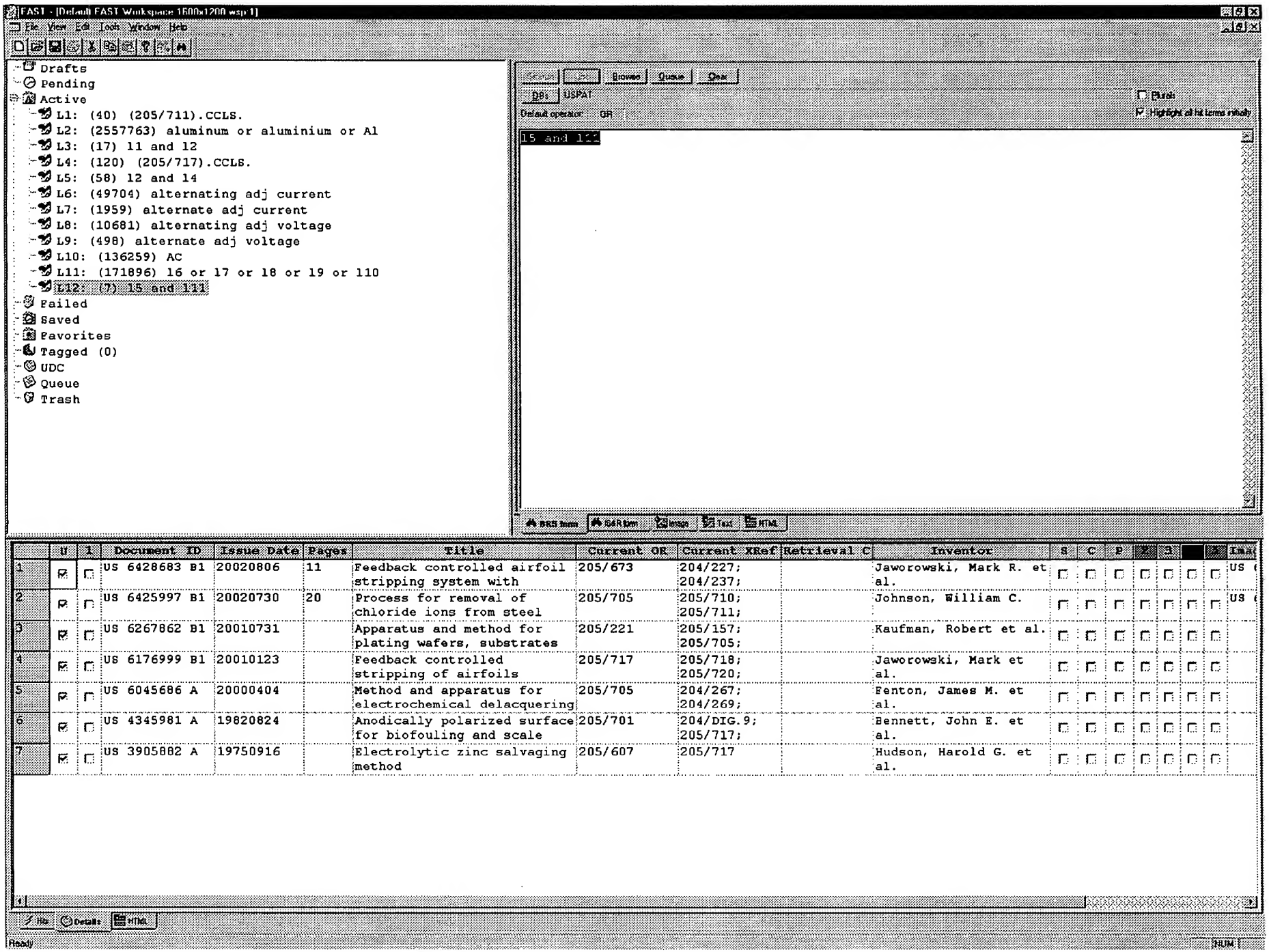
Primary Examiner—Howard S. Williams
Attorney, Agent, or Firm—Watson, Leavenworth, Kelton & Taggart

[57] ABSTRACT

In repairing by electrodeposition coating an end or top plate of an easy-open can along a score groove where the material metal has been exposed or along a deteriorated coated film chiefly on the reverse side of said end, a sine wave A.C. voltage is imparted as the bath voltage under the particular conditions, wherein the electric current of reverse direction is effectively utilized to effect penetration of the deteriorated coated part or removal of wax. In this case, special advantage can be obtained if an electrode made of the particular metal having an anodic oxidation film thereon is used.

7 Claims, 5 Drawing Figures





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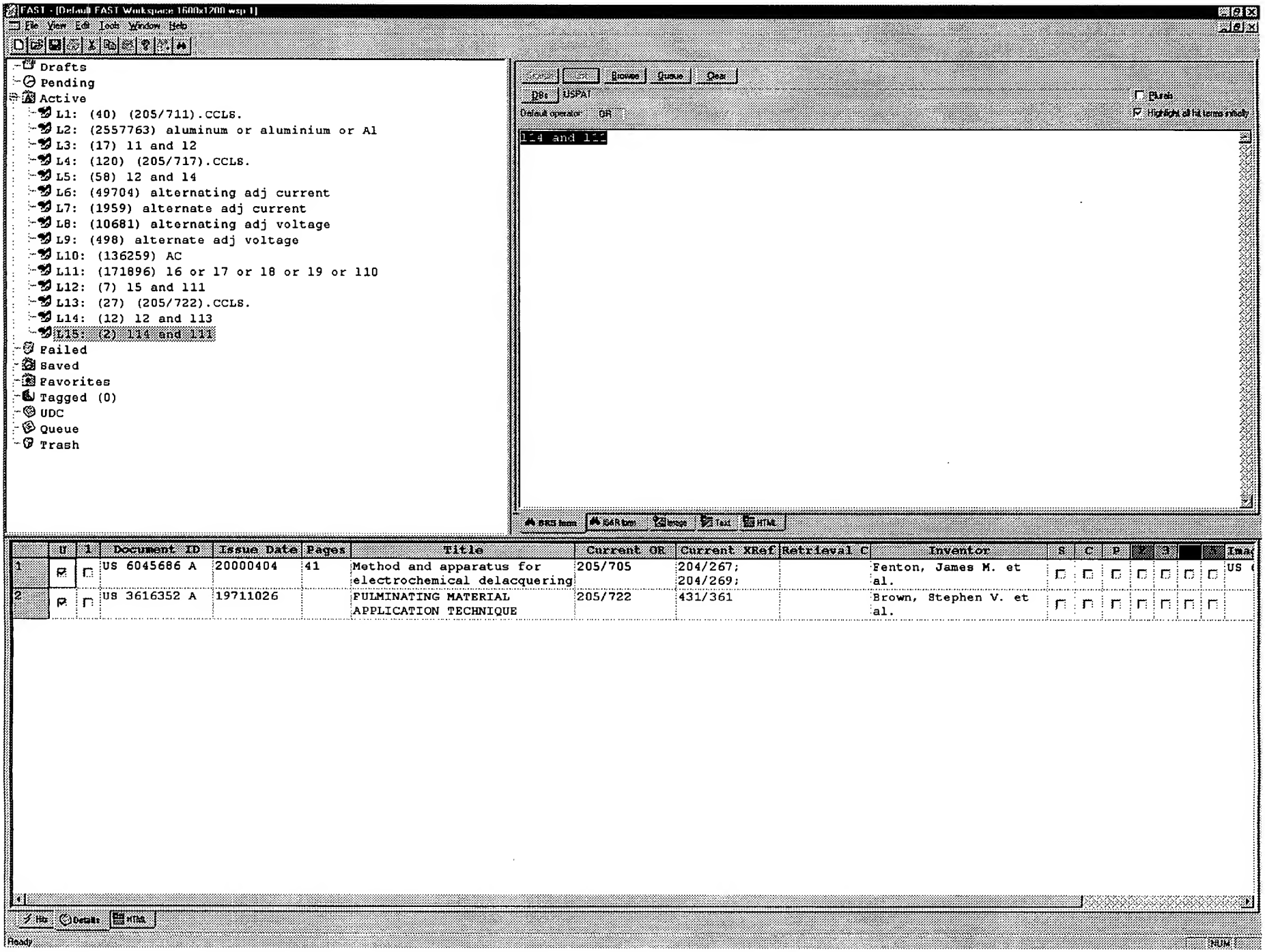
1	☑	☐	US 6428683 B1	20020806	11	Feedback controlled airfoil stripping system with	205/673	204/227; 204/237;		Jaworowski, Mark R. et al.	☐	☐	☐	☐	☐	☐	☐	US
2	☑	☐	US 6425997 B1	20020730	20	Process for removal of chloride ions from steel	205/705	205/710; 205/711;		Johnson, William C.	☐	☐	☐	☐	☐	☐	☐	US
3	☑	☐	US 6267862 B1	20010731		Apparatus and method for plating wafers, substrates	205/221	205/157; 205/705;		Kaufman, Robert et al.	☐	☐	☐	☐	☐	☐	☐	
4	☑	☐	US 6176999 B1	20010123		Feedback controlled stripping of airfoils	205/717	205/718; 205/720;		Jaworowski, Mark et al.	☐	☐	☐	☐	☐	☐	☐	
5	☑	☐	US 6045686 A	20000404		Method and apparatus for electrochemical delacquering	205/705	204/267; 204/269;		Fenton, James M. et al.	☐	☐	☐	☐	☐	☐	☐	
6	☑	☐	US 4345981 A	19820824		Anodically polarized surface for biofouling and scale	205/701	204/DIG.9; 205/717;		Bennett, John E. et al.	☐	☐	☐	☐	☐	☐	☐	
7	☑	☐	US 3905882 A	19750916		Electrolytic zinc salvaging method	205/607	205/717		Hudson, Harold G. et al.	☐	☐	☐	☐	☐	☐	☐	



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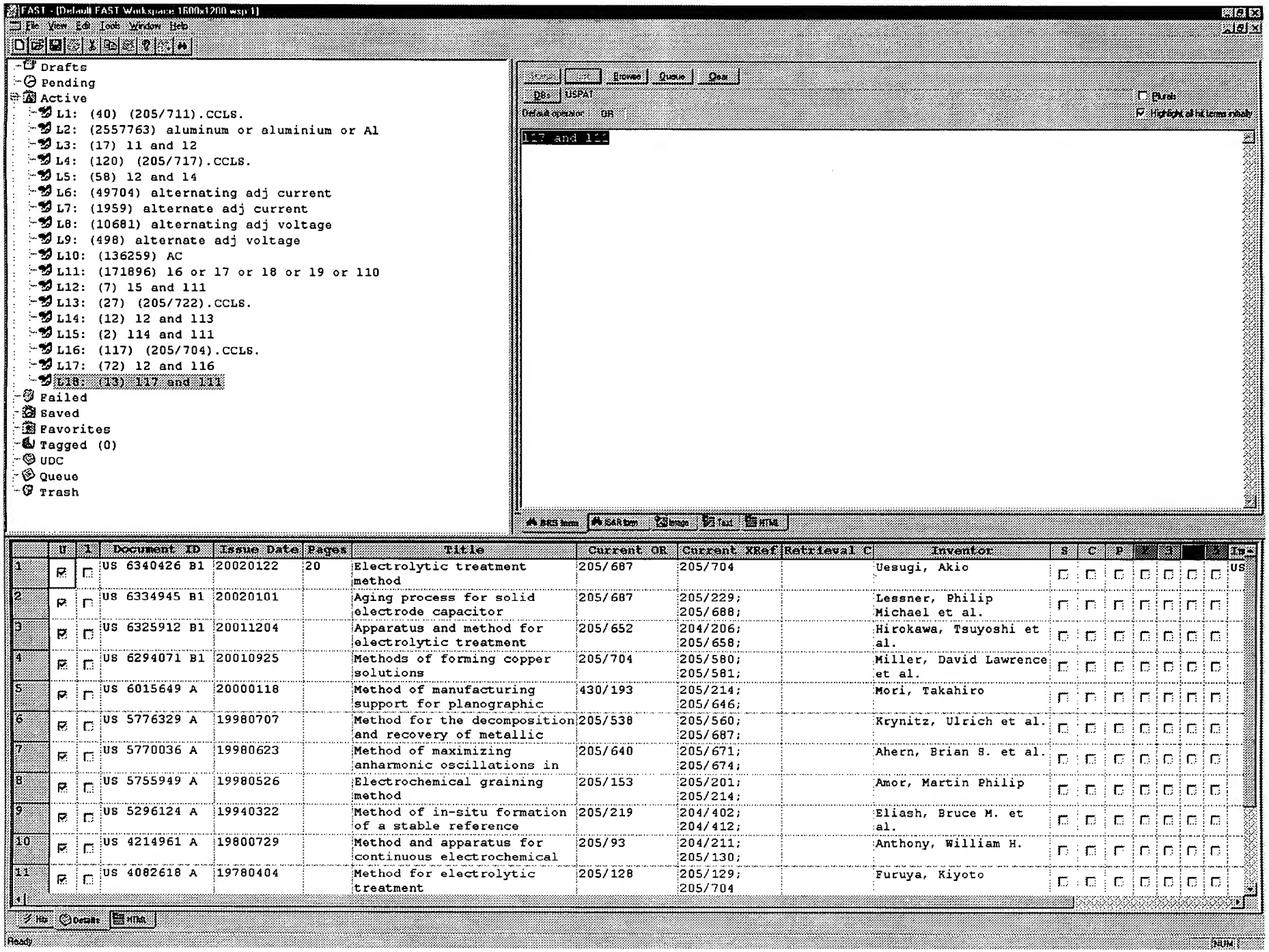
1	☑	☐	US 6045686 A	20000404	41	Method and apparatus for electrochemical delacquering	205/705	204/267; 204/269;		Fenton, James M. et al.	☐	☐	☐	☐	☐	☐	☐	US
2	☑	☐	US 3616352 A	19711026		FULMINATING MATERIAL APPLICATION TECHNIQUE	205/722	431/361		Brown, Stephen V. et al.	☐	☐	☐	☐	☐	☐	☐	



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1	☑	☐	US 6340426 B1	20020122	20	Electrolytic treatment method	205/687	205/704		Uesugi, Akio	☐	☐	☐	☐	☐	☐	☐	US
2	☑	☐	US 6334945 B1	20020101		Aging process for solid electrode capacitor	205/687	205/229; 205/688;		Lessner, Philip Michael et al.	☐	☐	☐	☐	☐	☐	☐	
3	☑	☐	US 6325912 B1	20011204		Apparatus and method for electrolytic treatment	205/652	204/206; 205/658;		Hirokawa, Tsuyoshi et al.	☐	☐	☐	☐	☐	☐	☐	
4	☑	☐	US 6294071 B1	20010925		Methods of forming copper solutions	205/704	205/580; 205/581;		Miller, David Lawrence et al.	☐	☐	☐	☐	☐	☐	☐	
5	☑	☐	US 6015649 A	20000118		Method of manufacturing support for planographic	430/193	205/214; 205/646;		Mori, Takahiro	☐	☐	☐	☐	☐	☐	☐	
6	☑	☐	US 5776329 A	19980707		Method for the decomposition and recovery of metallic	205/538	205/560; 205/687;		Krynitz, Ulrich et al.	☐	☐	☐	☐	☐	☐	☐	
7	☑	☐	US 5770036 A	19980623		Method of maximizing anharmonic oscillations in	205/640	205/671; 205/674;		Ahern, Brian S. et al.	☐	☐	☐	☐	☐	☐	☐	
8	☑	☐	US 5755949 A	19980526		Electrochemical graining method	205/153	205/201; 205/214;		Amor, Martin Philip	☐	☐	☐	☐	☐	☐	☐	
9	☑	☐	US 5296124 A	19940322		Method of in-situ formation of a stable reference	205/219	204/402; 204/412;		Eliash, Bruce M. et al.	☐	☐	☐	☐	☐	☐	☐	
10	☑	☐	US 4214961 A	19800729		Method and apparatus for continuous electrochemical	205/93	204/211; 205/130;		Anthony, William H.	☐	☐	☐	☐	☐	☐	☐	
11	☑	☐	US 4082618 A	19780404		Method for electrolytic treatment	205/128	205/129; 205/704		Furuya, Kiyoto	☐	☐	☐	☐	☐	☐	☐	

L18

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10	US 4214961 A	9					☑				USPAT
11	US 4082618 A	14					☑				USPAT
12	US 4019971 A	9					☑				USPAT
13	US 3962061 A	8					☑				USPAT

durability. It is to be noted that these properties are more excellent that when aluminum or aluminum alloy is coated in accordance with our copending application Ser. No. 484,985, filed July 1, 1974, in which aluminum or its alloy is electrolytically anodized with an aqueous solution of a water-soluble salt of the above-specified oxyacid and then electrophoretically coated with an aqueous organic coating composition not containing such water-soluble oxyacid salt. The reason why the aluminum or aluminum alloy treated in accordance with the present invention has such excellent properties has not been made clear yet, but it is supposedly attributable to the fact that in the electrolysis step of an aqueous solution of the water-soluble oxyacid salt, the oxyacid anions resulting from the dissociation of the oxyacid salt are adsorbed by the surface of aluminum or aluminum alloy, whereupon they release their charge and react with the boehmite layer or chemical conversion layer, thereby forming a new inorganic layer and that when the resulting aluminum or its alloy having the new layer is electrophoretically coated with an aqueous organic coating composition containing the water-soluble oxyacid salt, the oxyacid is adsorbed by the new layer first, then the oxyacid and the binder resin in mixture and the binder resin finally to produce a new complex coating layer having excellent compactness on the aluminum or its alloy with a high adhering strength.

Brief Summary Text - BSTX (10):

In practicing the process of this invention, aluminum or aluminum alloy is first subjected to usual pretreatment including degreasing and etching. Degreasing is conducted in usual manner, for example, by immersing aluminum or aluminum alloy in an acid such as nitric or sulfuric acid at room temperature. Similarly, etching is conducted in usual manner as by immersing aluminum or aluminum alloy in an alkali solution at a temperature of about 20.degree. to 80.degree.C. The aluminum or aluminum alloy thus pretreated is then subjected to boehmite treatment or chemical conversion treatment which is carried out by a conventional method.

alkali-e

Brief Summary Text - BSTX (11):

The boehmite treatment is usually conducted by contacting the pretreated aluminum or aluminum alloy with hot water or steam containing or not containing ammonia or amines. Examples of the amines usable are monoethanolamine, diethanolamine, triethanolamine, dimethylethanolamine and like water-soluble amines. Generally, about 0.1 to 5 parts by weight of amine or ammonia is used per 100 parts by weight of water. Use of such amine or ammonia increases the thickness of aluminum oxide layer produced by the boehmite treatment, but it is impossible to obtain an aluminum oxide layer having a thickness of more than about 1.0 .mu.. The aluminum or aluminum alloy is kept in contact with hot water or steam usually for about 5 to 60 minutes. The temperature of hot water to be used is usually in the range of 65.degree.C to boiling point, preferably 80.degree.C to boiling point and that of steam in the range of 100.degree. to 180.degree.C, preferably 130.degree. to 150.degree.C. Such contact is effected by methods heretofore employed, for example, by immersion or spraying.

Brief Summary Text - BSTX (13):

The aluminum or aluminum alloy thus subjected to boehmite or chemical conversion treatment is rinsed with water and then used as an electrode to conduct electrolysis in an aqueous solution of water-soluble salt of at least one oxyacid selected from the group consisting of silicic acid, boric acid

United States Patent [19]

Nikaido et al.

[11] **3,962,061**

[45] **June 8, 197[illegible]**

[54] **PROCESS FOR COATING ALUMINUM OR ALUMINUM ALLOY**

[75] Inventors: **Norio Nikaido; Shinji Shirai; Mototaka Ihashi; Suso Umemoto, all of Hiratsuka, Japan**

[73] Assignees: **Kansai Paint Company, Ltd.; Fuji Sasid Industries Limited, both of Japan**

[22] Filed: **Nov. 11, 1974**

[21] Appl. No.: **522,878**

[30] **Foreign Application Priority Data**

Nov. 20, 1973 Japan 48-131096

[52] U.S. Cl. 204/181

[51] Int. Cl.[2] C25D 13/06; C25D 13/10; C25D 13/20

[58] Field of Search 204/181

[56] **References Cited**

UNITED STATES PATENTS

2,868,702	1/1939	Brennan	204/28
3,290,235	12/1966	Gilchrist	204/181
3,622,473	11/1971	Ohta et al.	204/181
3,812,023	5/1974	Schardein et al.	204/5

FOREIGN PATENTS OR APPLICATIONS

2,160,428	6/1973	France	204/18

Primary Examiner—Howard S. Williams
Attorney, Agent, or Firm—Larson, Taylor and Hinds

[57] **ABSTRACT**

A process for coating an aluminum or aluminum allo comprising the steps of subjecting aluminum or alumi num alloy to boehmite treatment or chemical conver sion treatment, conducting electrolysis using the re sulting aluminum or aluminum alloy as an electrode i an aqueous solution of a water-soluble salt of at leas one oxyacid, and thereafter coating electrophoret ically the aluminum or aluminum alloy with an aque ous organic coating composition containing a binde resin and a water-soluble salt of at least one oxyacid t form a resin layer, said oxyacid contained in the aque ous solution and aqueous organic coating composition being at least one oxyacid selected from the grou consisting of silicic acid, boric acid, phosphoric acid molybdic acid, vanadic acid, permanganic acid, stan nic acid and tungstic acid.

25 Claims, No Drawings

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FAST Browser - [18 (13) 17 and 11] [US 3962061 A] [Tag: S] [Doc: 13/13 (SORTED)] [Format: KWIC]

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	Document ID	Pages	1	2	3	4	U	S	C	P	Kind Codes	Sou
8	US 5755949 A	13	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
9	US 5296124 A	6	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
10	US 4214961 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
11	US 4082618 A	14	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
12	US 4019971 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
13	US 3962061 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

permanganate [Mg(MnO.sub.4).sub.2 .6H.sub.2 O], strontium permanganate [Sr(MnO.sub.4).sub.2 .3H.sub.2 O], etc. The stannates include orthostannates and metastannates. Examples are potassium orthostannate (K.sub.2 SnO.sub.3.sup...3H.sub.2 O), lithium orthostannate (Li.sub.2 SnO.sub.3 .3H.sub.2 O), sodium orthostannate (Na.sub.2 SnO.sub.3 .3H.sub.2 O), magnesium stannate, calcium stannate, lead stannate, ammonium stannate, potassium metastannate (K.sub.2 O. 5SnO.sub.2 .4H.sub.2 O), sodium metastannate (Na.sub.2 O. 5SnO.sub.2 .8H.sub.2 O), etc. Examples of molybdates are orthomolybdates and metamolybdates. More specific examples are lithium molybdate (Li.sub.2 MoO.sub.4), sodium molybdate (Na.sub.2 MoO.sub.4), potassium molybdate (K.sub.2 MoO.sub.4), ammonium molybdate [(NH.sub.4).sub.6 Mo.sub.7 O.sub.24 .4H.sub.2 O], triethylamine molybdate, etc.

Brief Summary Text - BSTX (18):

According to this invention, the electrolysis is conducted in a conventional manner. For example, the aluminum or aluminum alloy and another electroconductive material used as electrodes are immersed in aqueous solution of the above-specified oxyacid salt, and electric current is applied between the electrodes. The electric current may be either direct current or alternating current. When direct current is used, the aluminum or aluminum alloy is to be the anode and when alternating current is used, the aluminum or aluminum alloy can be used either as anode or as cathode. The advantageous range of the electric voltage is from 5 to 300 volts for direct current, or from 5 to 200 volts for alternating current. The electric current is applied for more than 5 seconds. The temperature of the electrolytic solution is usually in the range between the solidifying point of the solution of the oxyacid salt and boiling point of the solution, preferably in the range of 20.degree. to 60.degree.C.

Brief Summary Text - BSTX (19):

According to this invention, the electrolytic operation can be conducted repeatedly twice or more times with an aqueous solution of the same oxyacid salt or with aqueous solution of different oxyacid salts. For example, electrolysis is conducted with an aqueous solution of silicate and then with the same aqueous solution of silicate, or first with an aqueous solution of silicate and subsequently with an aqueous solution of another oxyacid salt. When repeatedly carried out, the electrolysis also gives the resulting aluminum or aluminum alloy higher corrosion resistance than when it is conducted only once. Moreover, the electrolysis causes some water to undergo electrolysis to give off hydrogen gas in the form of bubbles. Consequently, the bubbling lowers the efficiency of the electrolytic operation. However, if the electrolysis is conducted repeatedly, the evolution of hydrogen gas is noticeably reduced as compared with the case wherein the electrolytic operation is conducted only once, assuring improved efficiency.

Brief Summary Text - BSTX (20):

After the electrolysis, the aluminum or aluminum alloy is rinsed with water and dried, whereby a thick layer of higher hardness and finer texture is formed. According to this invention, the dried product may further be heated at a temperature of about 150.degree. to 250.degree. C when desired to thereby increase the hardness of the coating.

United States Patent [19]

Nikaido et al.

[11] **3,962,061**

[45] **June 8, 1976**

[54] **PROCESS FOR COATING ALUMINUM OR ALUMINUM ALLOY**

[75] Inventors: **Norio Nikaido; Shinji Shirai; Mototaka Ihashi; Suso Umemoto,** all of Hirasuka, Japan

[73] Assignees: **Kansai Paint Company, Ltd.; Fuji Sashi Industries Limited,** both of Japan

[22] Filed: Nov. 11, 1974

[21] Appl. No.: **522,878**

[30] **Foreign Application Priority Data**

Nov. 20, 1973 Japan 48-131096

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[58] Field of Search 204/181

[56] **References Cited**

UNITED STATES PATENTS

2,868,702	1/1959	Brennan	204/28
3,290,235	12/1966	Gilchrist	204/181
3,622,473	11/1971	Ohta et al.	204/181
3,812,023	5/1974	Schardein et al.	204/5

FOREIGN PATENTS OR APPLICATIONS

2,160,428	6/1973	France	204/18

Primary Examiner—Howard S. Williams

Attorney, Agent, or Firm—Larson, Taylor and Hinds

[57] **ABSTRACT**

A process for coating an aluminum or aluminum alloy comprising the steps of subjecting aluminum or aluminum alloy to boehmite treatment or chemical conversion treatment, conducting electrolysis using the resulting aluminum or aluminum alloy as an electrode in an aqueous solution of a water-soluble salt of at least one oxyacid, and thereafter coating electrophoretically the aluminum or aluminum alloy with an aqueous organic coating composition containing a binder resin and a water-soluble salt of at least one oxyacid to form a resin layer, said oxyacid contained in the aqueous solution and aqueous organic coating composition being at least one oxyacid selected from the group consisting of silicic acid, boric acid, phosphoric acid, molybdic acid, vanadic acid, permanganic acid, stannic acid and tungstic acid.

25 Claims, No Drawings

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8	US 5755949 A	13										USPAT
9	US 5296124 A	6										USPAT
10	US 4214961 A	9										USPAT
11	US 4082618 A	14										USPAT
12	US 4019971 A	9										USPAT
13	US 3962061 A	8										USPAT

increase the hardness of the coating.

Brief Summary Text - BSTX (21):

The aluminum of aluminum alloy is thereafter electrophoretically coated with an aqueous organic coating composition containing a water-soluble salt of at least one oxyacid selected from the group consisting of silicic acid, boric acid, phosphoric acid, permanganic acid, vanadic acid, tungstic acid, molybdic acid, and stannic acid. The aqueous organic coating composition is prepared by adding the water-soluble salt to one of various aqueous electrophoretic coating compositions conventionally known. These known compositions generally comprise an aqueous medium and a water-soluble or water-dispersible binder resin dissolved or dispersed in the aqueous medium. The water-soluble salts to be added to the known aqueous electrophoretic coating compositions are various water-soluble salts of the specified oxyacids usable for the foregoing electrolytic operation.

Brief Summary Text - BSTX (24):

The electrophoretic coating operation is conducted in conventional manner. For example, the aluminum or aluminum alloy substrate to be coated is immersed into the electrophoretic coating composition in the bath and connected to the positive pole of a direct current. Another electroconductive material is immersed in the composition in the same bath and connected to the negative electrode and then direct current is applied between them. The voltage of said direct current is usually in the range of 30 to 400 volts.

Brief Summary Text - BSTX (27):

The process of this invention is applicable to various aluminum alloys such as Al --Si, Al --Mg, Al --Mn, Al --Si--Mg, and like alloys. The aluminum or aluminum alloy to be treated by the present process is not limited to plate or panel but may be of various shapes.

suggest add rolling

Detailed Description Text - DETX (1):

The process of this invention will be described below in greater detail with reference to examples and comparison examples, in which the percentages and parts are all by weight unless otherwise specified. In the examples aluminum panels serving as substrates are prepared by the method stated below, and electrolytic operation and electrophoretic coating operation are conducted according to the procedures stated below.

Detailed Description Text - DETX (3):

A substrate is prepared by degreasing and etching an aluminum alloy panel measuring 70 mm in width, 150 mm in length and 2 mm in thickness (consisting of 98.0% aluminum, 0.45% Si, 0.55% Mg and 1% others; JIS H 4100) according to the following procedure:

Detailed Description Text - DETX (11):

Into a plastic container measuring 10 cm in width, 20 cm in length and 15 cm in

United States Patent [19]

Nikaido et al.

[11] **3,962,061**

[45] **June 8, 197(**

[54] **PROCESS FOR COATING ALUMINUM OR ALUMINUM ALLOY**

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[22] Filed: Nov. 11, 1974

[21] Appl. No.: **522,878**

[30] **Foreign Application Priority Data**

Nov. 20, 1973 Japan 48-131096

[52] U.S. Cl. 204/181

[51] Int. Cl.[2] C25D 13/06; C25D 13/10; C25D 13/20

[58] Field of Search 204/181

[56] **References Cited**

UNITED STATES PATENTS

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FOREIGN PATENTS OR APPLICATIONS

2,160,428	6/1973	France	204/18

Primary Examiner—Howard S. Williams

Attorney, Agent, or Firm—Larson, Taylor and Hinds

[57] **ABSTRACT**

A process for coating an aluminum or aluminum allo comprising the steps of subjecting aluminum or alumi num alloy to boehmite treatment or chemical conver sion treatment, conducting electrolysis using the re sulting aluminum or aluminum alloy as an electrode i an aqueous solution of a water-soluble salt of at leas one oxyacid, and thereafter coating electrophoret ically the aluminum or aluminum alloy with an aque ous organic coating composition containing a binde resin and a water-soluble salt of at least one oxyacid t form a resin layer, said oxyacid contained in the aque ous solution and aqueous organic coating compositio being at least one oxyacid selected from the grou consisting of silicic acid, boric acid, phosphoric acid molybdic acid, vanadic acid, permanganic acid, stan nic acid and tungstic acid.

25 Claims, No Drawings

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8	US 5755949 A	13											USPAT
9	US 5296124 A	6											USPAT
10	US 4214961 A	9											USPAT
11	US 4082618 A	14											USPAT
12	US 4019971 A	9											USPAT
13	US 3962061 A	8											USPAT

respectively in place of sodium silicate used in the electrophoretic coating composition of Example 8. Aluminum substrates are treated in the same manner as in Example 8 except that these electrophoretic coating compositions are used respectively. The properties of the substrates thus obtained are listed in Table 3 below.

Detailed Description Text - DETX (53):

Aluminum substrates are treated in the same manner as in Example 1 except that the specified salts of oxyacids listed in Table 4 below are used respectively in place of sodium silicate used in the electrolytic bath of Example 1. The coating films obtained have substantially the same properties as one obtained in Example 1.

Detailed Description Text - DETX (55):

Aluminum substrates are treated in the same manner as in Example 8 except that the specified salts of oxyacids listed in Table 5 below are used respectively in place of potassium orthomolybdate used in the electrolytic bath of Example 8. The coating films obtained have substantially the same properties as one obtained in Example 8.

Detailed Description Text - DETX (57):

Aluminum substrates are treated in the same manner as in Example 1 except that the specified salts of oxyacids listed in Table 6 below are used respectively in place of potassium orthomolybdate used in the electrophoretic bath of Example 1. The coating films obtained have substantially the same properties as one obtained in Example 1.

Detailed Description Text - DETX (59):

Aluminum substrates are treated in the same manner as in Example 8 except that the specified salts of oxyacids listed in Table 7 below are used respectively in place of sodium silicate used in the electrophoretic bath of Example 8. The coatings obtained have substantially the same properties as one obtained in Example 8.

Claims Text - CLTX (1):

1. A process for coating an aluminum or aluminum alloy comprising the steps of subjecting aluminum or aluminum alloy to boehmite treatment or chemical conversion treatment, conducting electrolysis using the resulting aluminum or aluminum alloy as an electrode in an aqueous solution of a water-soluble salt of at least one oxyacid, and thereafter coating electrophoretically the aluminum or aluminum alloy with an aqueous organic coating composition containing a binder resin and a water-soluble salt of at least one oxyacid to form a resin layer, said oxyacid contained in the aqueous solution and aqueous organic coating composition being at least one oxyacid selected from the group consisting of silicic acid, boric acid, phosphoric acid, molybdic acid, vanadic acid, permanganic acid, stannic acid and tungstic acid.

Claims Text - CLTX (2):

monoethanolamine, diethanolamine, triethanolamine, dimethylethanolamine and like water-soluble amines. Generally, about 0.1 to 5 parts by weight of amine or ammonia is used per 100 parts by weight of water. Use of such amine or ammonia increases the thickness of aluminum oxide layer produced by the boehmite treatment, but it is impossible to obtain an aluminum oxide layer having a thickness of more than about 1.0 μ. The aluminum or aluminum alloy is kept in contact with hot water or steam usually for about 5 to 60 minutes. The temperature of hot water to be used is usually in the range of 65°C to boiling point, preferably 80°C to boiling point and that of steam in the range of 100° to 180°C, preferably 130° to 150°C. Such contact is effected by methods heretofore employed, for example, by immersion or spraying.

Generally, the chemical conversion treatment is conducted in conventional manner. Examples of the chemical conversion treatment are MBV method using sodium carbonate and sodium chromate, EW method using sodium carbonate, sodium chromate and sodium silicate, LW method using sodium carbonate, sodium chromate and sodium primary phosphate, Pylumin method using sodium carbonate, sodium chromate and basic chromium carbonate, Alrock method using sodium carbonate and potassium dichromate, Jirocka method using dilute nitric acid containing heavy metal or using a mixture of permanganic acid and hydrofluoric acid containing heavy metal, Pacz method using a mixture of sodium silicofluoride and ammonium nitrate which contains a nickel or cobalt salt, a method using manganese dihydrogenphosphate and manganese silicofluoride, and a method wherein acidic zinc phosphate, phosphoric acid and chromic acid are used, etc.

The aluminum or aluminum alloy thus subjected to boehmite or chemical conversion treatment is rinsed with water and then used as an electrode to conduct electrolysis in an aqueous solution of water-soluble salt of at least one oxyacid selected from the group consisting of silicic acid, boric acid, phosphoric acid, molybdic acid, vanadic acid, permanganic acid, tungstic acid and stannic acid.

The oxyacid salts to be used include various water-soluble salts of the above oxyacids with monovalent to trivalent metals, ammonia or organic amines. The silicates include orthosilicates, meta-silicates and disilicates and like polysilicates. Examples thereof are sodium orthosilicate, potassium orthosilicate, lithium orthosilicate, sodium metasilicate, potassium metasilicate, lithium metasilicate, lithium pentasilicate, barium silicate, ammonium silicate, tetramethanol ammonium silicate, trisethanol ammonium silicate, etc. The borates include metaborates, tetraborates, pentaborates, perborates, biborates, borate-hydrogen peroxide addition products and boroformates. Examples are lithium metaborate ($LiBO_2$), potassium metaborate (KBO_2), sodium metaborate ($NaBO_2$), ammonium metaborate, lithium tetraborate ($Li_2B_4O_7.5H_2O$), potassium tetraborate, sodium tetraborate, ammonium tetraborate [$(NH_4)_2B_4O_7.4H_2O$], calcium metaborate [$Ca(BO_2)_2.2H_2O$], sodium pentaborate ($Na_2B_{10}O_{16}.10H_2O$), sodium perborate ($NaBO_3.H_2O_2.3H_2O$), sodium borate-hydrogen peroxide addition product ($NaBO_2.H_2O_2$), sodium boroformate ($NaH_2BO_3.HCOOH.2H_2O$), ammonium biborate [$(NH_4)HB_4O_7.3H_2O$], etc. 52 The phosphates include orthophosphates, pyrophosphates and polymetaphosphates. Examples are potassium monobasic phosphate (KH_2PO_4), sodium pyro-

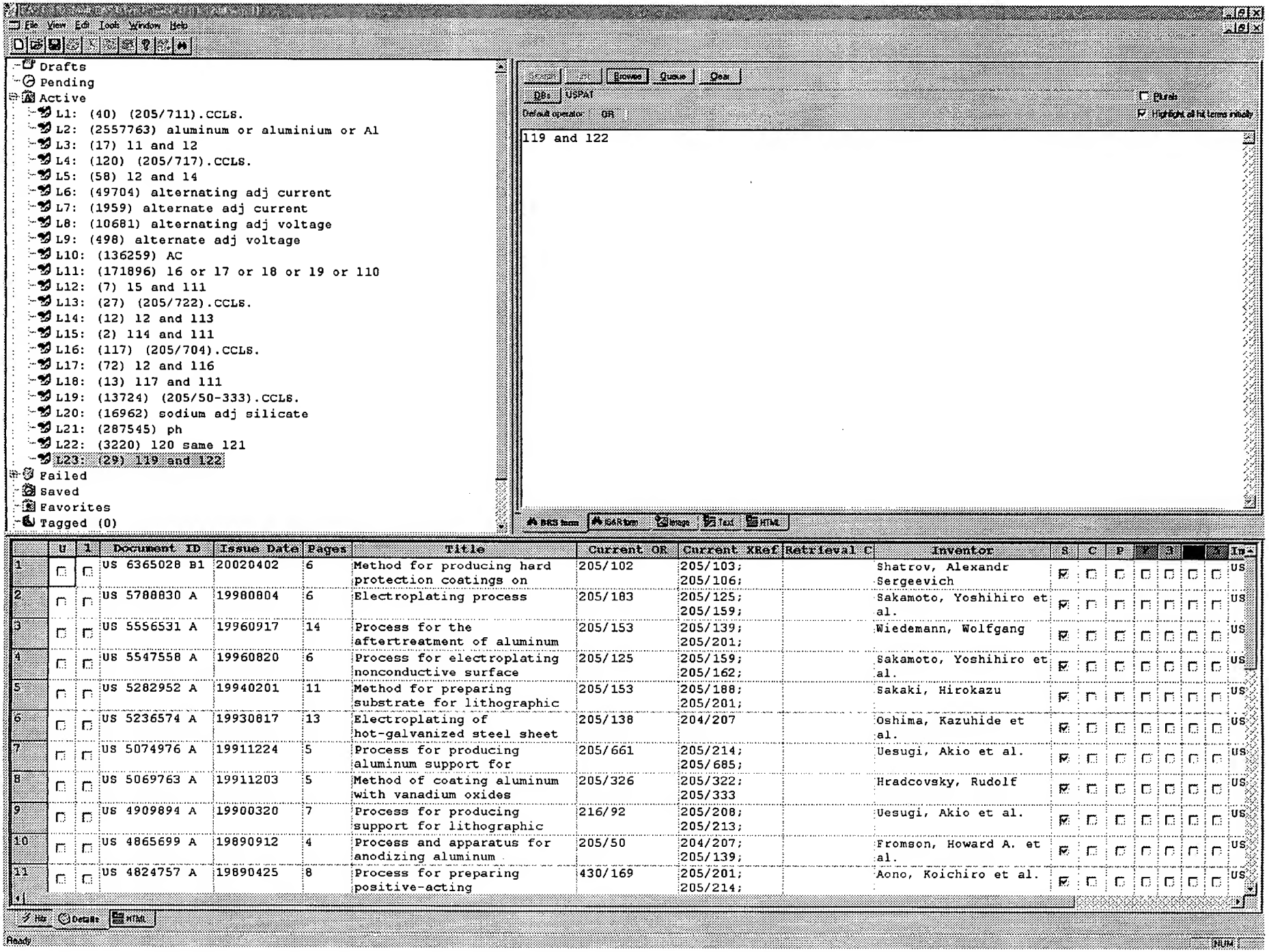
phosphate ($Na_4P_2O_7$), sodium metaphosphate ($NaPO_3$), aluminum hydrophosphate [$Al(H_2PO_4)_3$], etc. The vanadates include orthovanadates, metavanadates and pyrovanadates. Examples are lithium orthovanadate (Li_3VO_4), sodium orthovanadate (Na_3VO_4), lithium metavanadate ($LiVO_3.2H_2O$), .5 sodium metavanadate ($NaVO_3$), potassium metavanadate (KVO_3), ammonium metavanadate (NH_4VO_3) or [$(NH_4)_4V_4O_{12}$], sodium pyrovanadate ($Na_4V_2O_7$), etc. The tungstates include orthotungstates, metatungstates, paratungstates, pentatungstates and heptatungstates. Also employable are phosphorus wolframates, borotungstates and like complex salts. Examples are lithium tungstate (Li_2WO_4), sodium tungstate ($Na_2WO_4.2H_2O$), potassium tungstate (K_2WO_4), barium tungstate ($BaWO_4$), calcium tungstate ($CaWO_4$), strontium tungstate ($SrWO_4$), sodium metatungstate ($Na_6W_6O_{18}$), potassium metatungstate ($K_6W_6O_{18}.8H_2O$), sodium paratungstate ($Na_{10}W_{12}O_{46}$), ammonium pentatungstate [$(NH_4)_{10}W_{12}O_{42}.5H_2O$], ammonium heptatungstate [$(NH_4)_6W_7O_{24}.6H_2O$], sodium phosphowolframate ($2Na_2O.P_2O_5.12WO_3.18H_2O$), barium borotungstate ($2BaO.B_2O_3.9WO_3.18H_2O$), etc. Examples of permanganates are lithium permanganate ($LiMnO_4$), sodium permanganate ($NaMnO_4.3H_2O$), potassium permanganate ($KMnO_4$), ammonium permanganate [$(NH_4)MnO_4$], calcium permanganate [$Ca(MnO_4)_2.4H_2O$], barium permanganate [$Ba(MnO_4)_2$], magnesium permanganate [$Mg(MnO_4)_2.6H_2O$], strontium permanganate [$Sr(MnO_4)_2.3H_2O$], etc. The stannates include orthostannates and metastannates. Examples are potassium orthostannate ($K_4SnO_4.3H_2O$), lithium orthostannate ($Li_2SnO_3.3H_2O$), sodium orthostannate ($Na_2SnO_3.3H_2O$), magnesium stannate, calcium stannate, lead stannate, ammonium stannate, potassium metastannate ($K_2O.5SnO_2.4H_2O$), sodium metastannate ($Na_2O.5SnO_2.8H_2O$), etc. Examples of molybdates are orthomolybdates and metamolybdates. More specific examples are lithium molybdate (Li_2MoO_4), sodium molybdate (Na_2MoO_4), potassium molybdate (K_2MoO_4), ammonium molybdate [$(NH_4)_6Mo_7O_{24}.4H_2O$], triethylamine molybdate, etc.

Preferable among these oxyacid salts are those of alkali metals which generally have high water solubilities. Among the oxyacid salts enumerated above, silicates are preferable to use because they are economical and readily available. According to this invention these oxyacid salts are used singly or in admixture with one another.

The concentration of such oxyacid salt in its aqueous solution is usually about 0.1% by weight to saturation, preferably about 1.0% by weight to saturation, although variable with the kind of the oxyacid salt.

In the present invention, water-soluble salts of chromic acid can be used together with the abovementioned oxyacid salts, whereby the anti-corrosive property of the resulting coating is further improved. Examples of the chromates are lithium chromate ($Li_2CrO_4.2H_2O$), sodium chromate ($Na_2CrO_4.10H_2O$), potassium chromate (K_2CrO_4), ammonium chromate [$(NH_4)_2CrO_4$], calcium chromate ($CaCrO_4.2H_2O$) and strontium chromate ($SrCrO_4$).

According to this invention, the electrolysis is conducted in a conventional manner. For example, the aluminum or aluminum alloy and another electroconductive material used as electrodes are immersed in aqueous solution of the above-specified oxyacid salt, and electric current is applied between the electrodes.



	U	I	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C	P	3	In
1	☐	☐	US 6365028 B1	20020402	6	Method for producing hard protection coatings on	205/102	205/103; 205/106;		Shatrov, Alexandr Sergeevich	☑	☐	☐	☐	US
2	☐	☐	US 5788830 A	19980804	6	Electroplating process	205/183	205/125; 205/159;		Sakamoto, Yoshihiro et al.	☑	☐	☐	☐	US
3	☐	☐	US 5556531 A	19960917	14	Process for the aftertreatment of aluminum	205/153	205/139; 205/201;		Wiedemann, Wolfgang	☑	☐	☐	☐	US
4	☐	☐	US 5547558 A	19960820	6	Process for electroplating nonconductive surface	205/125	205/159; 205/162;		Sakamoto, Yoshihiro et al.	☑	☐	☐	☐	US
5	☐	☐	US 5282952 A	19940201	11	Method for preparing substrate for lithographic	205/153	205/188; 205/201;		Sakaki, Hirokazu	☑	☐	☐	☐	US
6	☐	☐	US 5236574 A	19930817	13	Electroplating of hot-galvanized steel sheet	205/138	204/207		Oshima, Kazuhide et al.	☑	☐	☐	☐	US
7	☐	☐	US 5074976 A	19911224	5	Process for producing aluminum support for	205/661	205/214; 205/685;		Uesugi, Akio et al.	☑	☐	☐	☐	US
8	☐	☐	US 5069763 A	19911203	5	Method of coating aluminum with vanadium oxides	205/326	205/322; 205/333		Hradcovsky, Rudolf	☑	☐	☐	☐	US
9	☐	☐	US 4909894 A	19900320	7	Process for producing support for lithographic	216/92	205/208; 205/213;		Uesugi, Akio et al.	☑	☐	☐	☐	US
10	☐	☐	US 4865699 A	19890912	4	Process and apparatus for anodizing aluminum	205/50	204/207; 205/139;		Fromson, Howard A. et al.	☑	☐	☐	☐	US
11	☐	☐	US 4824757 A	19890425	8	Process for preparing positive-acting	430/169	205/201; 205/214;		Aono, Koichiro et al.	☑	☐	☐	☐	US

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18	US 4576686 A	11							☑				USPAT
19	US 4561944 A	7							☑				USPAT
20	US 4387006 A	6							☑				USPAT
21	US 4331479 A	9							☑				USPAT
22	US 4252575 A	5							☑				USPAT
23	US 4225398 A	11											USPAT

US-PAT-NO: 4252575

DOCUMENT-IDENTIFIER: US 4252575 A

TITLE: Producing hydrous oxide of controlled thickness on aluminum capacitor foil

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Detailed Description Text - DETX (6):

The foil may subsequently be treated with a dilute aqueous phosphate of pH 5 to 7 solution or a sodium silicate solution partly neutralized to pH 7 to 12, preferably 10 to 11, by tartrate as described in copending patent application Ser. No. 35,145 filed May 2, 1979, to further increase capacitance upon anodization.

Detailed Description Text - DETX (10):

In order to compare the behavior of hydrous oxide films prepared by a conventional boiling water treatment and by the borate treatment of the present invention, aluminum foil etched for low-voltage use was contacted with boiling water or with a boiling solution of 6.0 g/l boric acid adjusted to pH 6 with borax. After formation of the hydrous oxide films, the foils were contacted with a boiling sodium silicate solution for 7.5 min. The foils were anodized to 60 V in a 0.1% aqueous ammonium dihydrogen phosphate solution.

Current US Cross Reference Classification - CCXR (1):

205/201

how to neutralize with acid to get pH down

United States Patent [19]

Bernard

[11] 4,252,575

[45] Feb. 24, 1981

[54] PRODUCING HYDROUS OXIDE OF CONTROLLED THICKNESS ON ALUMINUM CAPACITOR FOIL

[75] Inventor: Walter J. Bernard, Williamstown, Mass.

[73] Assignee: Sprague Electric Company, North Adams, Mass.

[21] Appl. No.: 68,302

[22] Filed: Aug. 9, 1979

[51] Int. Cl.[3] C23F 7/06

[52] U.S. Cl. ..... 148/6.27; 204/33; 204/38 A

[58] Field of Search ..... 148/6.27; 204/33, 38 A

[56] References Cited

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W. J. Bernard and J.J. Randall, J. Electrochem Soc., 108#9, pp. 822-825, (Sep. 1961).

Primary Examiner—Ralph S. Kendall

Attorney, Agent, or Firm—Connolly and Hutz

[57] ABSTRACT

The thickness of a hydrous oxide layer on aluminum capacitor foil is controlled by producing the oxide in a hot, dilute borate solution with a pH of about 6, prior to anodization of the foil.

10 Claims, 2 Drawing Figures

FAST Browser - I 23 (29) 19 and 22 | US 5282952 A | Tag: S | Doc: 5/29 (SORTED) | Format: KWIC

File Edit View Tools Window Help

	Document ID	Pages	1	2	3	4	u	s	c	p	Kind Codes	Sou
1	US 6365028 B1	6						☑				USPAT
2	US 5700830 A	6						☑				USPAT
3	US 5556531 A	14						☑				USPAT
4	US 5547558 A	6						☑				USPAT
5	US 5282952 A	21						☑				USPAT
6	US 5236574 A	13					☑					USPAT
7	US 5074976 A	5					☑					USPAT

US-PAT-NO: 5282952

DOCUMENT-IDENTIFIER: US 5282952 A

TITLE: Method for preparing substrate for lithographic printing plates, substrate for lithographic printing plates prepared by the method and presensitized plate comprising the substrate

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Detailed Description Text - DETX (43):

The resulting aluminum plates each was immersed, at 100.degree. C. for 30 seconds, in an aqueous solution of H.sub.2 SO.sub.4 having a pH value of 1, 3 or 5 respectively (Comparative Examples 6 to 8), deionized water of pH 6 (Comparative Example 9), an aqueous solution of KOH having a pH value of 8, 9, 10, 11, 12 (Examples 6 to 10) or 13 (Comparative Example 10). Further, the plates were anodized at a current density of 10 A/dm.sup.2 and 30.degree. C. in an electrolyte comprising 170 g/l of sulfuric acid and 8 g/l of Al.sup.3+ ions to form an anodic oxide layer in an amount of 3 g/m.sup.2. Then these plates were immersed in a 2.5% aqueous solution of JIS No. 3 sodium silicate at 70.degree. C. for 10 seconds. Light-sensitive Composition I having the composition defined in Example 1 was applied onto these substrates in an amount of 1.0 g/m.sup.2 (weighed after drying).

Detailed Description Text - DETX (56):

Each PS plate thus prepared was imagewise exposed to light from a 3 KW metal halide lamp at a distance of 1 m for 50 seconds through a transparent positive film in a vacuum printing frame and then developed with a 5.26% aqueous solution (pH =12.7) of sodium silicate whose molar ratio: SiO.sub.2 /Na.sub.2 O was 1.74.

Current US Original Classification - CCOR (1):

205/153

Current US Cross Reference Classification - CCXR (1):

205/180

Current US Cross Reference Classification - CCXR (2):

205/201

Current US Cross Reference Classification - CCXR (3):

205/213

[11] Patent Number: 5282952

Sakaki [45] Date of Patent: Feb. 1, 1994

[54] **METHOD FOR PREPARING SUBSTRATE FOR LITHOGRAPHIC PRINTING PLATES, SUBSTRATE FOR LITHOGRAPHIC PRINTING PLATES PREPARED BY THE METHOD AND PRESENSITIZED PLATE COMPRISING THE SUBSTRATE**

[75] Inventor: Hirokazu Sakaki, Shizuoka, Japan

[73] Assignee: Fuji Photo Film Co., Ltd., Minami-Ashigara, Japan

[21] Appl. No.: 745,414

[22] Filed: Aug. 15, 1991

[30] Foreign Application Priority Data

Aug. 16, 1990 [JP] Japan 2-215980
Oct. 12, 1990 [JP] Japan 2-276795
Oct. 12, 1990 [JP] Japan 2-276796

[51] Int. Cl.[5] C25D 11/16
[52] U.S. Cl. 205/153; 205/186; 205/201; 205/213; 205/921
[58] Field of Search 205/153, 159, 127, 213, 205/214, 921, 188, 201

[56] References Cited

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Primary Examiner—John Niebling
Assistant Examiner—William T. Leader
Attorney, Agent, or Firm—Burns, Doane, Swecker & Mathis

[57] **ABSTRACT**

A method for preparing a substrate for lithographic printing plates comprises the steps of forming a hydrated oxide layer on the surface of an aluminum plate and then anodizing the plate in a sulfuric acid electrolyte; a lithographic printing plate comprises the substrate; and the substrate per se prepared by the method is also disclosed herein.

The presensitized plate for use in making lithographic printing plates which comprises the substrate has very high sensitivity which makes the presensitized plate applicable to new exposure methods and is capable of being developed with a developer of an aqueous solution system. The light-sensitive layer and the substrate of the presensitized plate are strongly adhered to one another and, therefore, the resulting lithographic printing plate has excellent printing durability and good printability.

11 Claims, No Drawings

FAST Browser - [23: (29) 19 and 22 | US 4865699 A | Tag: 5 | Doc: 10/29 (SORTED) | Format: KWIC]

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	Document ID	Pages	1	2	3	4	U	S	C	P	Kind Codes	Sou
5	US 5282952 A	11						☑				USPAT
6	US 5236574 A	13						☑				USPAT
7	US 5074976 A	5						☑				USPAT
8	US 5069763 A	5						☑				USPAT
9	US 4909894 A	7						☑				USPAT
10	US 4865699 A	4						☑				USPAT
11	US 4824757 A	8					☑					USPAT

US-PAT-NO: 4865699

DOCUMENT-IDENTIFIER: US 4865699 A

TITLE: Process and apparatus for anodizing aluminum

---------- KWIC ---------

Detailed Description Text - DETX (8):

Three stainless steel electrodes were placed in an insulated silicating cell containing 3% sodium silicate at a pH of 11.7--Philadelphia Quartz Start Brand. The electrodes were immersed approximately 12 inches in the solution approximately 6 inches from the moving aluminum web. The electrodes were attached as anodes 32 to a rectifier 24" as shown in FIG. 2. Three cathodes 34 in the cell 10' were also attached to the same rectifier 24".

Current US Original Classification - CCOR (1):

205/50

Current US Cross Reference Classification - CCXR (2):

205/139

Current US Cross Reference Classification - CCXR (3):

205/334

Current US Cross Reference Classification - CCXR (4):

205/83

United States Patent [19]

Fromson et al.

[11] Patent Number: 4,865,699

[45] Date of Patent: Sep. 12, 1989

[54] **PROCESS AND APPARATUS FOR ANODIZING ALUMINUM**

[76] Inventors: Howard A. Fromson, 15 Rogues Ridge Rd., Weston, Conn. 06880; Robert F. Gracia, P.O. Box 104, Old Turnpike Rd., Woodstock, Conn. 06282

[21] Appl. No.: 211,425

[22] Filed: Jun. 24, 1988

[51] Int. Cl.⁴ C25D 11/04

[52] U.S. Cl. 204/28; 204/207

[58] Field of Search 204/28, 206, 207

[56] References Cited

U.S. PATENT DOCUMENTS

3,865,700 2/1975 Fromson 204/28

Primary Examiner—T. M. Tufariello

Attorney, Agent, or Firm—Pelfe & Lynch

[57] ABSTRACT

Anodizing current is prevented from being carried downstream by an aluminum web after anodizing in a continuous anodizing process. An anode is placed in a post-treatment cell containing an electrolyte which is electrically connected to a cathode in the anodizing cell via a source of DC current which is independent of the source of anodizing current. The web polarity is switched from positive to negative and a voltage greater than the exist voltage of the web is applied to prevent anodizing current from being carried downstream in the web after anodizing.

11 Claims, 1 Drawing Sheet